DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS
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BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 48

AN AIR ANALYZER FOR DETERMINING THE FINENESS OF CEMENT

BY

J. C. PEARSON, Assistant Physicist

and

W. H. SLIGH, Aid

Bureau of Standards

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AN AIR ANALYZER FOR DETERMINING THE FINENESS OF CEMENT

By J. C. Pearson and W. H. Sligh

CONTENTS

I. INTRODUCTION—GENERAL DISCUSSION OF ELUTRIATION METHODS IN THE MECHANICAL ANALYSIS OF CEMENT

The fineness of cement is one of its most important and at the same time one of its most indefinite characteristics. Its importance is recognized in the tendency toward higher fineness requirements in cement specifications and in the general belief that the finer a cement is ground the greater its cementing value. Its indefiniteness is due to the almost universal method of determining fineness by means of the No. 100 and No. 200 sieves. The inadequacy of these sieves is evident when one considers that at least 75 per cent of the total cement is required to pass the No. 200 sieve. It is also well known that a considerable portion of the cement passing the No. 200 sieve is comparatively inert because the larger particles in this portion are still too coarse to be readily acted upon by water.

What is needed, therefore, is some means of determining the amount of hydraulically active material in cement. At the present time, however, we do not know what size of particles should be regarded as the upper limit of active material, and thus the logical mode of procedure would seem to be first to develop some method of separating still further the "fines" from the No. 200 sieve, and then to establish a dividing line between inert and active particles.

The terms "flour" and "impalpable powder" are frequently used to designate the hydraulically active material. It is believed preferable to limit these terms to that very fine portion of cement beyond the finest perceptible grit, which probably does not constitute all of the hydraulically active material.

An endeavor has been made at the Bureau of Standards to develop an analyzer capable of giving a mechanical analysis of that portion of cement passing the No. 200 sieve. Such an analysis it appears can best be made by elutriation methods, in which the desired separations are accomplished by washing or blowing the fine material from the coarse. Two types of apparatus need thus be considered, one employing a liquid and the other a gas. In common with most investigators of this subject we believe that a gas or air analyzer is particularly adapted to cement and possesses distinct advantages over the liquid type. The disadvantages of the latter appear to consist largely in the inconvenience of recovering the separated material from the liquid and the difficulty of separating the very fine particles. On the other hand, the liquid type is probably better adapted to making a number of separations in one operation, which compensates to some extent its aforementioned disadvantages. While our own experience is limited to the air analyzer, we believe that either type is adapted to cement analysis, and provided certain essential features are embodied in their construction, satisfactory elutriators of very different forms and dimensions can be devised.

Our experience has indicated that the most important factors in elutriator design are:

1. The apparatus should insure as far as possible a constant velocity and uniform stream lines in the fluid as it passes through the separating chamber.

2. The fluid should not be appreciably retarded by the resistance of the material under examination or by constrictions or obstructions in that part of the apparatus beyond the separating chamber unless the amount of such retardation can be determined by pressure gages or compensated by special devices.

3. All particles of the material should be completely and continuously exposed to the action of the fluid, so that any which are capable of passing through the separating chamber may have every opportunity to do so.

4. The separating chamber should have no places of lodgment for material.

5. The apparatus should be capable of separating fair-sized samples, preferably 25 g. or more. Thus, representative samples

are more nearly insured and the percentage error in the separations is reduced.

More difficult than the mere design and construction of a satisfactory elutriator is its calibration or standardization. The mechanical analysis involves separations in terms not only of percentages but also of sizes, and the determination of the size of very small and irregularly shaped particles can be most readily accomplished by averaging microscopic measurements on a large number. Herein lies the one great obstacle to the furtherance of elutriation methods, for if the fineness curves of different cements are to be at all comparable the sizes of separation must be determined with considerable accuracy. It is not sufficient simply to state that the average diameter of a lot of particles which lie on the dividing line between two fractions is so many thousandths of an inch, or so many hundredths of a millimeter, but it is necessary to define what is meant by the diameter of an irregular particle, and to state explicitly how the diameters are measured. For example, if the diameters of a number of particles as seen in the microscope are measured in one direction without regard to their orientation (which is perhaps the easiest and most direct method) the average diameter so determined will be very approximately the mean of the average length and breadth of the particles as seen in the microscope. This average diameter, however, is greater than the cube root of the product of the average length, breadth, and thickness of the particles, which is generally taken to be the true mean diameter and the true index of "size" in the sense of volume. But the measurement of three diameters of microscopic particles is exceedingly laborious, and the most one can expect for practical purposes is to establish some reduction factor which will give the true mean diameter when applied to the simpler measurements.

Still another condition enters which magnifies the differences in these systems of measurement: The separation of powders by flotation methods obviously depends on the "floating power" of the particles, a property which presumably bears as close a relation to shape and surface condition of the particles as to their actual size and weight, and we may therefore anticipate a greater range in diameters of particles so separated than, for example, in uniform sieve separations.

II. SCOPE OF THE WORK

The chief aim of the work in developing an air analyzer for cement at the Bureau of Standards has been, first, the construction of an apparatus designed on the principles already set forth, and second, the calibration of the apparatus in such manner as to give reliable mechanical analyses of different brands of cement. After more than two years' investigation sufficient progress has been made to warrant publication of the results thus far obtained, but it must be admitted that only the more important phases of the entire problem have as yet been considered. The present status of the investigation may be best described by stating that the analyzer is capable of separating cement into almost any desired fractions, of which the quantities can be determined with the same accuracy as those obtained by sieving and the sizes of separation can be determined with fair accuracy sufficient for purposes of comparison.

III. REVIEW OF PREVIOUS TYPES OF ELUTRIATORS WHICH HAVE BEEN USED OR PROPOSED FOR THE MECHANICAL ANALYSIS OF CEMENT

A cooperative attempt to develop a standard elutriator for cement was undertaken several years ago by a committee of the International Association for Testing Materials. The problem of this committee was set forth as the "Determination of the simplest method for the separation of finest particles in Portland cement." Reports of progress were made by the chairman of the committee in the proceedings of the association for 1906, 1909, and 1912, and these reports were supplemented by papers presented by members who participated in the investigation. In these reports and papers a systematic study of the problem has been made, and in them will be found a more complete discussion of several of the types of apparatus briefly described in the following paragraphs.

1. GOREHAM'S FLOUROMETER

The following description of this apparatus (Fig. 1) is given in the reports of the Brussels Congress of the International Association for Testing Materials:[1]

It consists chiefly of two tubes *R* and *r*, the latter inside the former. The outer zinc tube *R* has a diameter of 8 cm and is closed at the top and tapered at the bottom. It fits air-tight into a tapered receptacle in which the cement to be tested is placed. The tube *r*, placed inside *R*, is carried air-tight through the cover of *R* and reaches as far as the narrowest part of the vessel *K*. The end piece is notched so as to prevent the narrow space between the two tubes from being choked with cement. Tube *R* carries at half its height an uptake tube which ends inside a large receptacle *B*. The air is driven through the inner tube from an aerometer *W* at a fixed pressure and passes through the notches; then it strikes against the tapered sides of the vessel *K* and stirs up the cement, of which the receptacle contains about 40 grams. The flour is carried off by the current of air, and the greater portion settles in receptacle *B*. At the end of the test the cement residue is weighed.

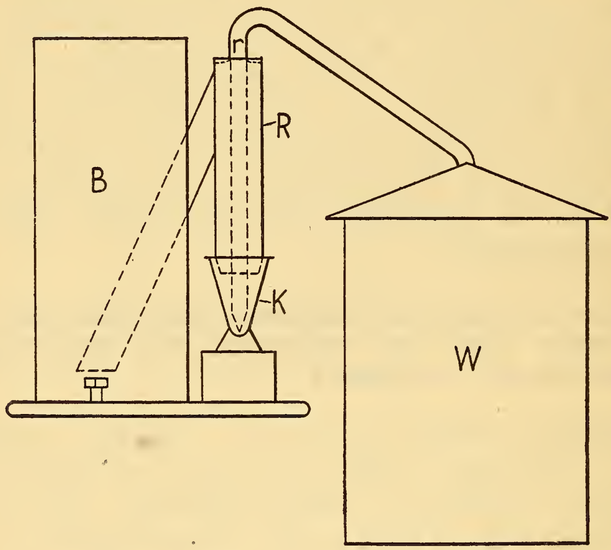

Fig. 1.—*Goreham flourometer*

This apparatus has more good points in its favor than a number of later types and is to be recommended especially for its simplicity. The main criticism of its operation would appear to be

[1] Determination of a Uniform Method for the Separation of the Finest Particles in Portland Cement by Liquid and Air Processes. Report by M. Gary, chairman of committee 30, Brussels Congress, Int. Ass'n Test. Mat'ls, 1906.

a considerably retarded air stream at the beginning of the separation, the air flowing more freely as the residue diminishes. A less important point is the fact that the "fines" leave the separating chamber *R* from one side of the latter only, whereas the delivery should preferably be symmetrical around the tube. The latter should also be high enough to avoid the removal of excessively heavy particles by impulses from the bottom.

2. THE GARY-LINDNER APPARATUS

The Gary-Lindner apparatus is perhaps the best-known elutriator for cements that has been devised, and its description, as given by Gary, one of its inventors, is also to be found in the International Association Reports of the Brussels Congress, 1906.[1a]

This apparatus, which also depends on the action of a current of air, but arriving at a more complete granulation, is shown in Fig. 2. It consists of three good-sized glass tubes *a*, which are connected with each other and which end in glass funnels. Small glass tubes for conveying the air, and extending almost to the bottom, are fused into the funnel tubes. Twenty grams of the powder to be tested, which must be thoroughly dried beforehand, are placed in the first funnel *I*. An air blast at a pressure of 100 mm (height of water) is then blown in. The air pressure for each funnel is regulated by glass cocks and is read off on a U gage. The funnels *I*, *II*, and *III* come successively into operation. Finally a portion of the powder will remain in each funnel, and the flour, which arrives as dust at the end of the third glass tube, is caught in the receptacle *IV*. The process admits of a granulation of four fractions of the cement powder, which are not separated exclusively with regard to the size of the grains, but nevertheless show relative characteristic differences.

Fig. 3 is a photograph of one of the Gary-Lindner separators, which gives a somewhat better idea of its proportions. Our experience with this apparatus bore out the experience of others who had tried it in this country in that we were unable to obtain satisfactory separations. Perhaps better results might have been obtained if detailed instructions had been available for the operation of the apparatus, but after some study of its behavior we became convinced that this type of elutriator was not adapted to quantitative separations. By referring to the essential features of a satisfactory elutriator enumerated on page 5, it will be seen that the Gary-Lindner separator fails particularly in the fourth requirement, and to some extent in the first and second. Blown material is certain to lodge at the junctions of the tubes *a*, for it

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[1a] See note 1, p. 8.

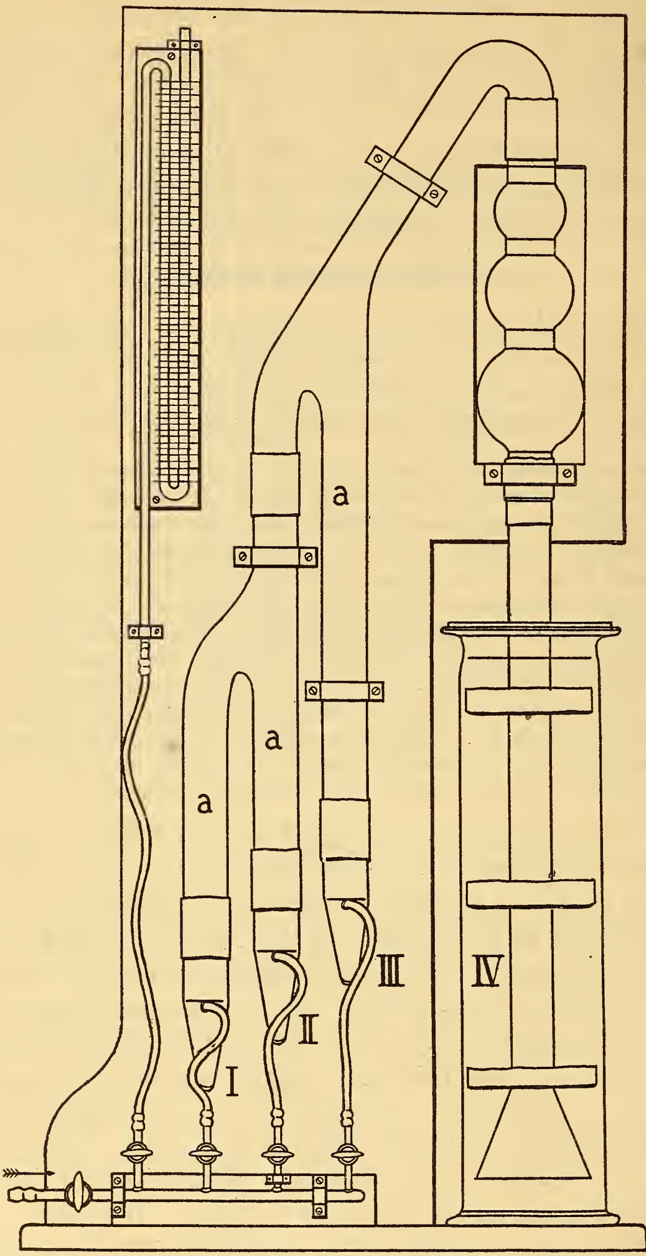

FIG. 2.—*Gary-Linder apparatus*

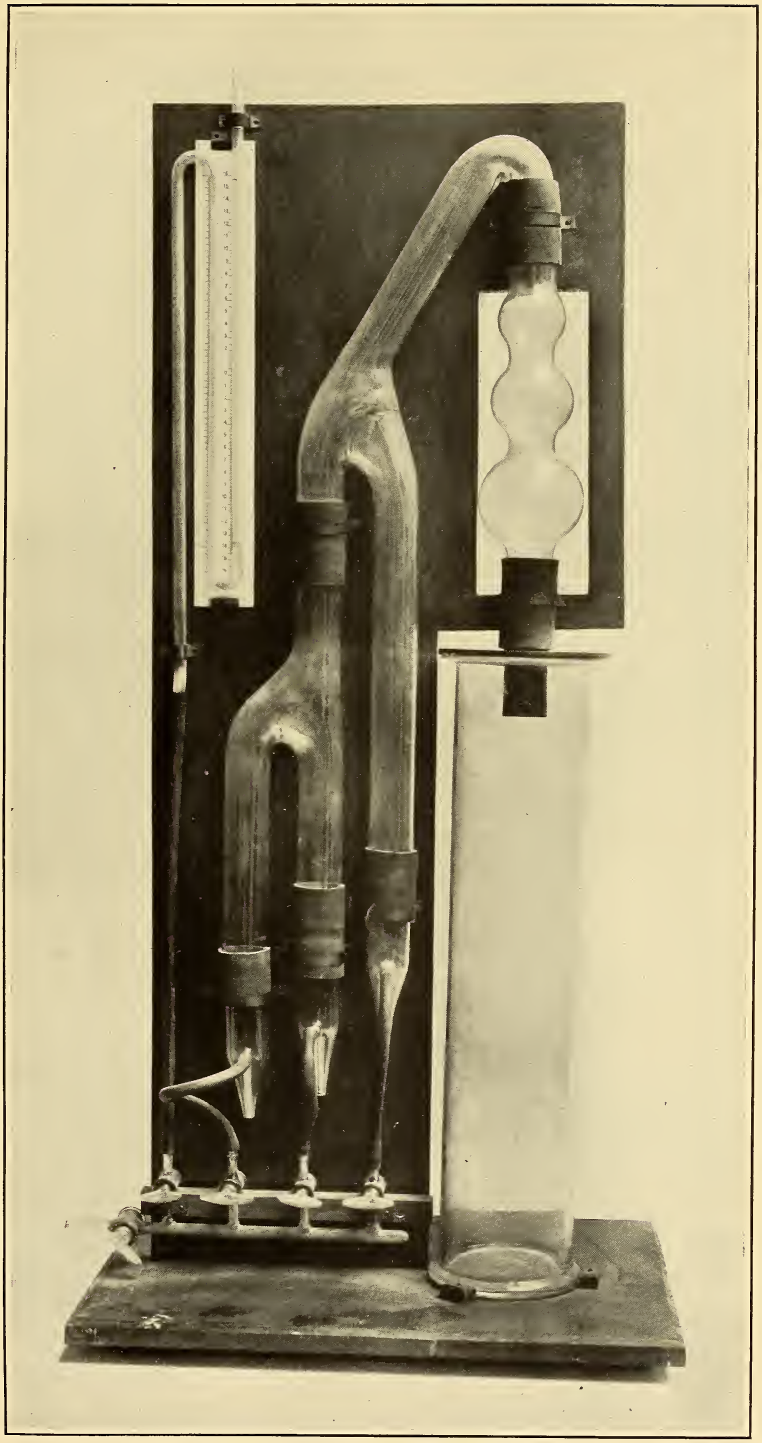

FIG. 3.—*Gary-Lindner apparatus*

is practically impossible to avoid shelves at these points, and the inclined tube connecting with the vessel *IV* will effectually prevent any pure separation of material to the right and left of this point. It has been our experience that any departure from the vertical in the walls of the separating tubes is an undesirable feature, tending to destroy the purity of the separations and adding to the difficulties of keeping the apparatus clean.

3. CUSHMAN AND HUBBARD'S AIR ELUTRIATOR

This apparatus was designed primarily for the separation of rock powders, and is described in the Journal of the American Chemical Society for 1907.[2]

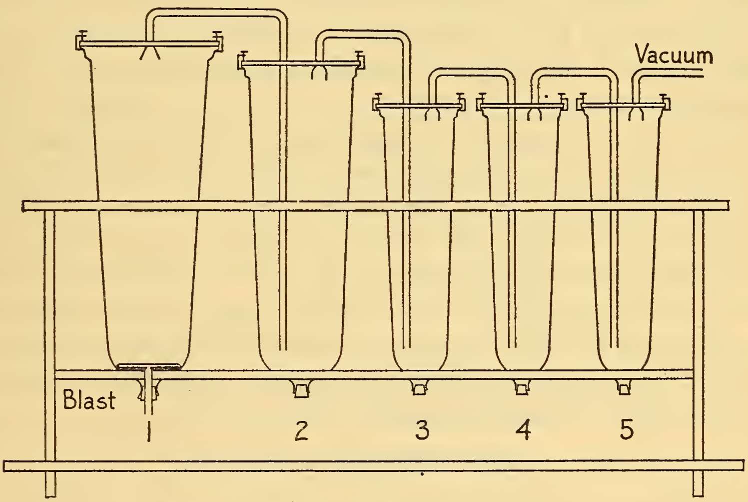

FIG. 4.—*Cushman-Hubbard elutriator*

The apparatus consists of five percolating jars set in a wooden frame and connected by tubes of glass passing through close-fitting caps tightly clamped to the jar tops, as shown in Fig. 4. The first jar is of 3 gallons capacity, the second 2 gallons, and the third, fourth, and fifth, each 1 gallon. In the bottom of No. 1 is placed a flat spiral tube closed at one end but with a number of very small openings through small jets soldered into the upper surface of the spiral at an angle of about 30°. The open end of the spiral passed through a tightly fitting rubber stopper inserted in the neck of the jar and is connected to the source of air supply. An inverted funnel tube whose stem passes through another rubber stopper fitted in the cap at the top of the jar is connected to a glass tube which passes in a similar manner nearly to the bottom of No. 2. An inverted thistle tube connects No. 2 with No. 3 in like manner, and so

[2] Cushman and Hubbard, Air Elutriation of Fine Powders, Jour. Am. Chem. Soc., 29, p. 589, 1907.

on through Nos. 3 and 4, the exit tube of No. 5 being connected to a vacuum. It is possible to tie pieces of fine linen lawn over the mouths of the inverted thistle tubes, but in our own work this has not been done except in the final exit in No. 5. Rubber stoppers close the necks of the jars and are removed only when it is desired to draw off the charges of powder which have accumulated during a run. A charge of oven-dried powder not exceeding 1 kg is placed in jar No. 1. Blast and vacuum are then turned on and adjusted so that a steady stream of air passes through the powder with sufficient force to raise a dense white cloud which assumes a vortex motion as it ascends, owing to the arrangement of the air jets. The heavier particles continually fall in a ring near the walls of the vessel, where they build up until caved in by the air jets, while the lighter particles are carried into No. 2 through the funnel tube. Here the heavier portions are retained while the lighter pass into No. 3, and so on, the cloud in each succeeding jar becoming less dense. If properly adjusted there is but little loss in No. 5, although the powder here is so fine that no trace of grit can be noticed when it is placed between the teeth.

The Cushman and Hubbard elutriator was used mainly to give only very rough quantitative separations of powders and is obviously incapable of the pure separations which are required for the mechanical analysis of cement. Even if the principle of this elutriator could be adapted to making a number of separations in one operation, the order of the separating jars must evidently be from small to large, since the accumulation of successive residues composed of smaller and smaller particles must depend upon diminishing rather than increasing air velocity through the system. The chief difficulties to be overcome in the construction of such an apparatus are the avoidance of places of lodgment for material in the tubes connecting the separating chambers and an adequate control of the air flow in all parts of the system.

4. THE PETERSEN APPARATUS

This apparatus is a modified form of Schöne's levigating funnel, originally used with liquids, but adapted for and used with air by Petersen in his cement investigations.[3] A diagram of the essential parts of the apparatus is shown in Fig. 5 and its description is quoted below:

The air enters at *a*, passes through the cockpiece *h*, the rubber tube *d*, the bent glass tube *e*, and the rubber tube *f*, up into the funnel *T*, where the levigation takes place. The cockpiece is situated at the lateral tube with the cock *c* in connection with the pressure gage *m*. The sections of the tubes *b* and *c* are of the same diameter

[3] Determination of the Simplest Method for the Separation of the Finest Particles in Portland Cement by Liquid and Air Processes. Appendix to the committee's report, M. Petersen, 5th Cong. Int. Ass'n Test. Mat'ls, Copenhagen, 1909.

as the clear opening in the corresponding glass tubes, so that the air meets with no resistance. By means of the cock *b* the air current can be effectually and quickly cut off, when the residue is removed from the funnel after the levigation. Oil of turpentine, which is sufficiently mobile, is applied in the pressure gage. The air current required for the experiments is produced by means of a centrifugal blower which is placed in connection with the cockpiece *h* by means of a rubber tube. A slide valve is inserted directly in front of the rubber tube, by means of which the air current can be regulated so that a constant height of pressure can be maintained in the pressure gage.

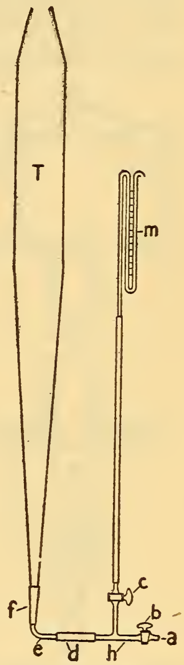

Fig. 5.—*Petersen apparatus*

Elsewhere in the above paper a device is described for introducing the sample of cement into the top of the separating funnel, and another for testing the purity of the separations, but no mention is made of a dust collector of any sort. If the top end of the separating tube was left open during the course of the experiments, the apparatus was certainly in its best working condition, except for the practical disadvantage of exposing everything in the immediate neighborhood to the settling dust. Only 5 g of cement constituted a charge, however, an amount which yields a proportionately small quantity of dust, but the charge is undesirably small for two reasons. The first is that samples of this size may not represent the true average granular composition of the cements as a whole, and second, very small errors in determining the residues, either from accident or from uncertainty in the stopping point of the operation, which in our experience must be determined by the rate of loss and not by the duration of the experiment, will be relatively large when expressed in percentages. Another objection to this "up-blast" type of elutriator is the tendency of the cement to choke in the throat of the separating funnel, which causes fluctuations of considerable magnitude in the air pressure of the supply and requires automatic regulation of the latter. Petersen states that on this account he was unable to use more than 5 g of cement in his analyses.

Whether the residue in the funnel be large or small, it is always being lifted by the incoming air stream, and consequently exerts more or less retardation upon the air delivery, even if the pressure of the supply be automatically regulated. Theoretically,

then, the retardation, and consequently the velocity, of the air through the separating chamber is dependent on the amount of residue left in the funnel; that is, it may be different for different cements, even though the air supply be maintained at constant pressure. This reduction in air delivery may or may not be negligible, depending on the amount of cement used, the sharpness of the cone of the separating funnel, and the normal air pressure used in the experiments, but it can hardly be disregarded. Petersen may have included this effect in his term "choking," but it should be distinguished from the peculiar behavior of the cement which is frequently observed when a sample too large for the air stream to handle readily is first introduced into an elutriator of this type. When the operation is proceeding properly, the whole mass of cement quivers and "teeters" as the air passes through it, gradually removing the fine dust. Very frequently, however, the cement lodges in the cone, the air forces an open "pipe" up through it and blows freely through the separating funnel, leaving the cement undisturbed in the bottom. Continual tapping of the funnel, shutting off and turning on the air supply, and other means must be resorted to of coaxing the cement into action. This contrary behavior of cement is well known to those who have worked with the Petersen type of apparatus, and it constitutes one of the chief objections to the "up-blast" elutriator, for it is a difficulty which increases enormously with the fineness of the separation.

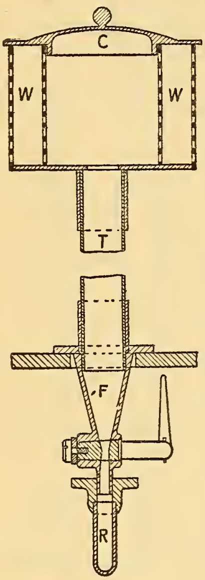

Fig. 6.—*Griffin-Goreham flourometer*

5. THE GRIFFIN-GOREHAM STANDARD FLOUROMETER

This apparatus is described in a number of treatises on Portland cement, the following description being taken from R. K. Meade's Portland Cement:[4]

This apparatus consists of two parts, an aerometer or blower and the apparatus proper, or flourometer. The blower consists of the customary bell and water tank and is merely used to furnish a constant supply of air to the apparatus. The flourometer itself (Fig. 6) consists of a long brass tube *T*, resting upon a stand. The sepa-

[4] Portland Cement, by R. K. Meade, 2d ed., 512 pp. The Chemical Publishing Co., Easton, Pa. 1911

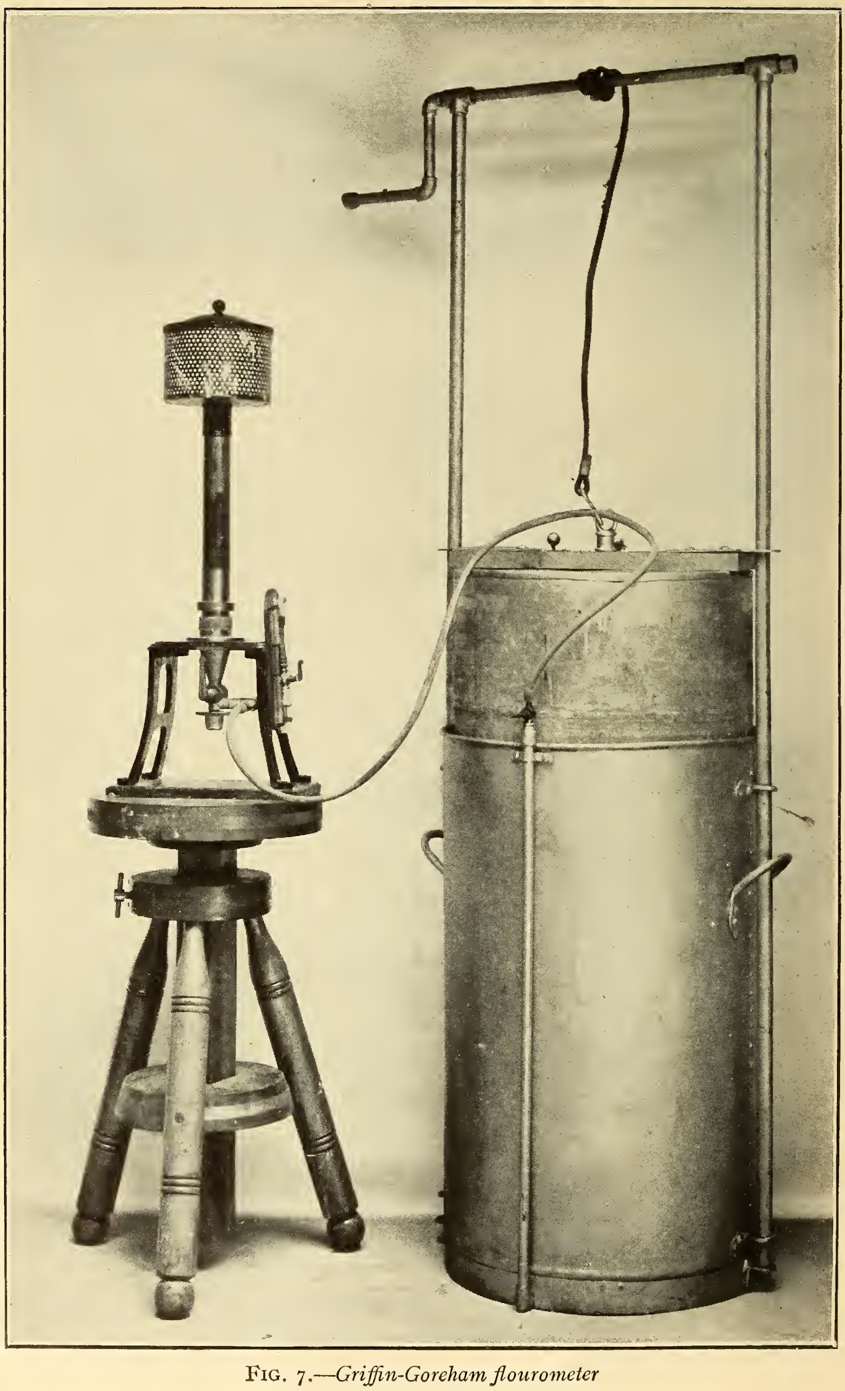

FIG. 7.—*Griffin-Goreham flourometer*

ration of the coarse and fine particles takes place in this. The tube is surmounted by a double-walled dome *W*, covered with a top *C*. The walls of this dome are perforated, and the spaces in between them, *W*, are filled with cotton. This serves to catch all dust and prevents it being blown into the laboratory. The lower part of the brass tube *T* terminates in a cone-shaped brass casting *F*, which rests upon the stand. A three-way stopcock provided with a pointer to show the direction of the opening is placed at the lower end of the cone and beneath this a glass tube *R*, which serves to catch the coarse particles. The sample of cement should be dried for an hour at 110°. The pointer of the stopcock should be at right angles to the brass tube *T*. The tube *T* is removed, and about 1 gram of the cement is then introduced into the funnel *F*. The bell of the aerometer is now raised to its full extent and the air pressure noted. The pointer of the stopcock is next turned parallel with the tube and the air allowed to blow through the apparatus for 10 minutes. At the end of this time the air pressure is turned off, when the coarse particles from which the cement has been separated drop into the receptacle *R*. This residue is weighed, and the difference of course is flour.

A Griffin-Goreham flourometer was purchased by the Bureau when the work in air analysis was first taken up, and the results of experiments with this apparatus led us to believe that a greater range in separations, as well as better control of the entire process, was desirable. The flourometer serves the purpose of illustrating in a simple manner the principle of air analysis of cements; aside from this its usefulness is limited. In passing it may be pointed out that all the objections which have been brought forward to the Petersen apparatus apply in even greater degree to the Griffin-Goreham flourometer, and the latter was soon discarded in our laboratory in favor of a more suitable design.

6. THE THOMPSON CLASSIFIER

An elutriator using kerosene has been constructed by G. W. Thompson for the study of paint pigments. The apparatus has also been used to some extent for cement analysis and was described in a paper before the American Society for Testing Materials in 1910.[5]

The plan of the Thompson classifier is shown in Fig. 8, the principle of its operation depending on a flow of kerosene at constant head into the upper cone, the overflow passing successively into the larger cones below. The material under examination is first diffused in kerosene so that the particles are completely

[5] The Classification of Fine Particles According to Size, G. W. Thompson; Proc. Amer. Soc. for Test. Mat'ls, 10, p. 601; 1910.

separated and transferred into the upper cone. The glass tube supplying the kerosene for the separation is then lowered into the cone until it nearly touches the apex, and the current is started. The operation is then practically automatic until the classification is completed. At the end of the operation the residue in each cone is washed into clock glasses with kerosene, allowed to settle, decanted, and washed two or three times with ether. It is then dried and weighed.

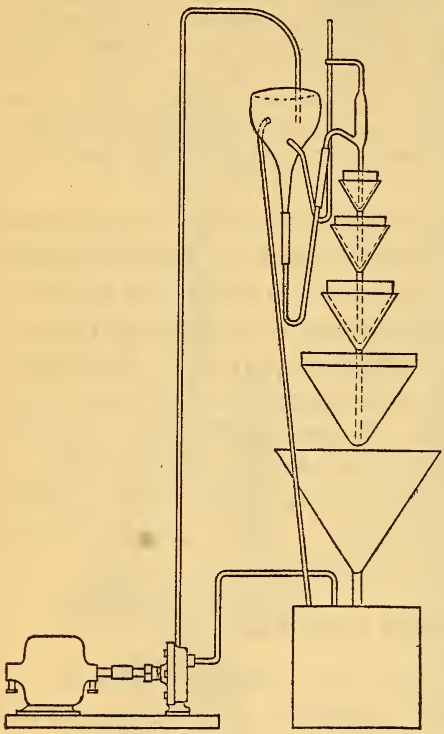

Fig. 8.—*Thompson classifier*

The Thompson classifier possesses the distinct advantage of making several separations in one operation, a time-saving feature which has not yet been successfully introduced into any type of air separator. On the other hand, certain disadvantages suggest themselves, which can only be established as decided objections when more data are available. It has been shown, for example, that the separations of pigments by this apparatus are not wholly reliable, owing either to their incomplete diffusion in the liquid when first introduced into the upper cone, or their tendency to agglomerate in some later stage of their preparation for microscopic examination. There appeared to be less of this difficulty, however, with certain siliceous and crystalline materials,[6] and it is possible that cement would be free from this objection. Another feature of this apparatus which may have a considerable effect on the purity of the separations is the possibility of irregular currents in the cones. The rise of the liquid in all the cones takes place with

[6] Report of Subcommittee J of Committee D-1 on the Testing of White Paints, P. H. Walker, chaiman; Proc. Amer. Soc. Test. Mat., 13, pp. 406-447; 1913.

rapidly diminishing speed, until at the point of overflow all vertical components of the flow have disappeared. It is almost inconceivable, therefore, that there should be uniform stream lines in the cones, in consequence of which the sizes of separation must be less sharply defined than it would be possible and desirable to have them. But it is impossible to state how serious this objection may be in cement analyses. From our experience we should anticipate that even if fractions showing satisfactory quantitative agreement could be obtained in repeated trials, the determination of the size of separation would be more or less uncertain.

7. FERET'S AIR SIFTER

This apparatus is the same in principle as Petersen's apparatus and is described in a paper by R. Feret before the Sixth Congress of the International Association for Testing Materials, New York, 1912.[7]

The paper is devoted to a theoretical study of an apparatus represented diagrammatically in Fig. 9, which is explained by the following excerpt:

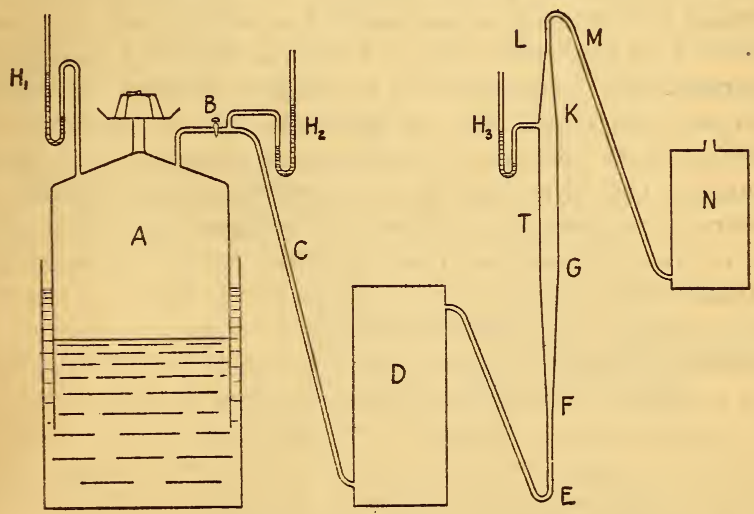

FIG. 9.—*Feret's air sifter*

A is a recipient in which air is maintained at a constant pressure; it communicates by a tap *B*, with a system of pipes *C E*, which lead the air into the sifting chamber

[7] Air Sifting as a Method for the Quantitative Determination of the Finest Particles Contained in Pulverulent Materials, R. Feret; Proc. I. A. T. M., Sixth Congress, Section II, XV2, 1912.

proper T, where the separation between two consecutive classes of grains takes place, the air escaping to the atmosphere through the pieces $L\ M\ N$. D represents a device which may be placed in front of the separator T, in which the carrying off of the grains is to be ascertained, and consisting, for example, of a drying column and other separators. N represents all the various pieces of the apparatus in the rear of the separator T, such as further separators and receivers for the finer dust carried away. H_1, H_2, and H_3 are the three pressure gages showing the excess h_1, h_2, h_3 of air pressure at the corresponding points over atmospheric pressure H.

Feret's studies led him to the almost self-evident conclusion that the limiting size of grains removed by apparatus of this type, or, as we shall hereafter designate it, the size of separation, depends solely upon the flow, and it is the quantity of air delivered which must be controlled and maintained constant. He states further that the regulation of the flow is incorrectly based on the constancy of the pressure of the air supply h_1, unless special precautions are taken to insure the constancy of the resistances in the entire apparatus, but depends rather on the constancy of the pressure in the top of the separating funnel h_3, together with the constancy of the resistance beyond the latter point.

Without going into the derivation of the foregoing result, and assuming it to be true, we are inclined to believe that the general scheme of an apparatus such as Feret has described and studied is unnecessarily complicated as a practical analyzer for cement. Thus we have found that an apparatus can be constructed in which the back pressure in the separating chamber (h_3 in Feret's apparatus) can be reduced to an entirely negligible quantity, at the same time preventing the escape of all dust except that which may be so attenuated as to be invisible. A single pressure gage attached to an air reservoir with an automatic regulator has been found adequate to insure the constancy of the air flow in the separating chamber, at least to such a degree that the variations have a negligible influence on the separations in comparison with other uncertainties. A practical objection has also been pointed out that is common to all the up-blast types of separators operating at comparatively low pressures, viz, such types are adapted only to the examination of undesirably small samples.

8. MACKEY'S APPARATUS

One of the most important investigations ever undertaken to determine the value of fine grinding of Portland cement was carried out some years ago at the University of Kansas by Dr. J. F. Mackey. The results of this investigation became available to us through the courtesy of the Ash Grove Lime & Portland Cement

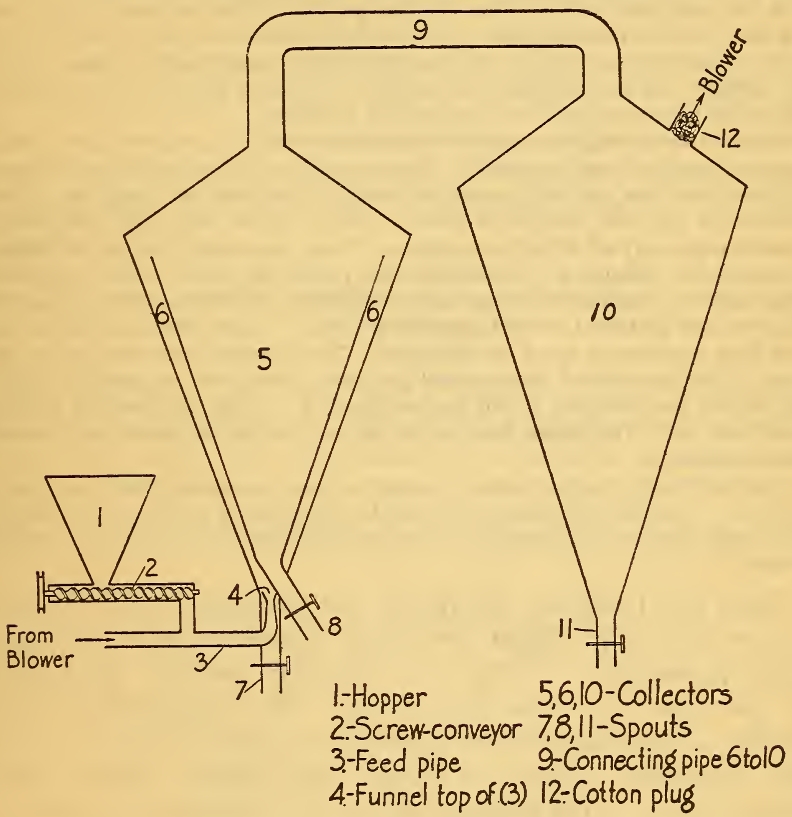

FIG. 10.—*Mackey's apparatus*

Co., of Kansas City, Mo., who established the fellowship under which the work was done.[8] A drawing of Dr. Mackey's apparatus is shown in Fig. 10, and his description of it is as follows:

The large separator was used to obtain large quantities of what is ordinarily termed "flour of cement." The cement to be separated was placed in the hopper (1) and

[8] The Fine Grinding of Portland Cement and the Effect of Hydrated Lime on Portland Cement. Dr. J. F. Mackey.

by means of a screw conveyor (2) fed into the pipe (3), through which a current of air from a blower was passing. By this means the cement was carried into the separators (5), (6), and (10). The arrangement of these conical-shaped separators is shown in the diagram. The cross-sectional area between (5) and (6) gradually increased from the bottom upward, causing the velocity of the air carrying the cement to gradually decrease as it ascended between (5) and (6). Practically all of the coarser particles were dropped in this portion of the apparatus. Any of the finer powder adhering to the walls of this part of the apparatus was removed by gentle tapping.

In the open space above (5) considerable portions of finer material dropped out and were removed from the collector by means of the spout at (8). The finest portions were carried over into (10), which served merely as a collector for these particles. The particles still in suspension were caught in the cotton plug (12) or were carried back to the blower and again sent through the separators.

When the sample had been sent through the apparatus, the products of the various containers were collected separately. The material in (10) was quite free from grit. The whole machine was then cleaned out and all of the materials except the portion collected in (10) sent through as before. Two or three such operations usually removed practically all of the finest powder. There was a small loss due to leakage. In general the contents of (6) contained that portion too coarse to pass a 220-mesh sieve, together with a small amount of finer material; the contents of (5) passed the 220 sieve and contained a small quantity of "flour." The contents of (5) and (6) were then separated in small air elutriators. The separators were arranged in batteries of 8 or 10 and dried air or natural gas blown through under constant velocity. The smaller particles were blown out first, grade C. Then the particles grade B were blown out. The residue was then sieved, and the portion passing the 220-mesh sieve was grade A.

I was not able to obtain a complete separation into a number of fractions with one operation. A special separation was used for each size of particle. The method used, while laborious, nevertheless gave results sufficiently accurate for the purpose in view.

From the foregoing description we understand that Mackey did not expect to obtain very sharp separations with the apparatus represented in Fig. 10, although perhaps satisfactory for the tests that were carried out. The arrangement of the smaller air elutriators is not described in sufficient detail to warrant discussion. Suffice it to say that Mackey obtained very fair agreement in the amounts of the four fractions below the No. 220 sieve. Extensive series of tests were made on these fractions, and valuable information was obtained, but we are concerned for the present only with the identification of the various fractions. Unfortunately Mackey reported only the average apparent diameters of the particles of the different grades, from which it is impossible to deduce the limiting sizes. Without knowing the limiting sizes—that is, the sizes of separation—we can not

derive the mechanical analysis curves, and the microscopic measurements are therefore of little assistance in identifving the separations.

In conclusion of this review of previous types of elutriators, it may be pointed out that there has apparently been no successful attempt to define separations of very fine material in absolute terms, in consequence of which the fractions obtained by one apparatus can not be compared with those obtained by any other. In the work which has been done at the Bureau it has been our particular endeavor to define our separations without reference to the apparatus. In this attempt we have met with a very fair degree of success, and we hope that the methods described in the following pages will encourage a more general use of elutriation processes.

IV. GRADUAL DEVELOPMENT OF A NEW FORM OF AIR ANALYZER

Our experience in air separation of cements really began in the early part of 1912, when a Griffin-Goreham flourometer was purchased and preliminary experiments were made on a number of cements. After a few trials it was believed that this apparatus could be improved and adapted to the examination of larger samples by using a source of air at considerably higher pressure than could be obtained by means of the aerometer furnished with the apparatus. Accordingly, further experiments were made using air from the compressed-air supply in the laboratory at about 2 pounds pressure per square inch. This change necessitated a longer stack, which was constructed of a piece of glass tubing about 1½ inches in diameter and 5 feet long, the upper end being bent in a sharp semicircle to facilitate attaching a dust-collecting sack. Varying the air flow by means of constricted pieces of tubing introduced into the supply line, we were able with this modification to obtain a number of fractions in the fine portion of the cement. In this form the apparatus presented three serious drawbacks—(1) it was noticed that a large amount of fine material collected in the bend of the stack, a considerable portion of which could only be dislodged by taking down the stack and running a cleaner

through it; (2) large pressure variations in the air supply were of frequent occurrence, due partly to the variations in the supply pressure and partly to the choking effect of the cement; (3) there was always trouble from choking and lodging of the cement in the cone at the bottom, this being especially annoying when a small current of air was used for the purpose of blowing out only the very fine particles.

The first of these objections was partly overcome by replacing the stack with another in which an offtake tube was sealed onto the vertical part of the stack at the sharpest possible angle. This stack is still in use as the separating chamber of a later type of analyzer, but the shelf formed at the junction of the offtake, although very narrow, is nevertheless a source of slight error on account of the tendency of material which should be carried off into the collector to lodge there and partly fall back into the coarse residue. It will be shown later that this difficulty can be very easily avoided, yet it constitutes the one glaring fault of the Gary-Lindner apparatus, and in our opinion precludes the use of the latter for quantitative separations of the required purity.

The pressure variations, so far as they were due to variations in the air supply, were avoided by procuring a small May-Nelson two-ring pump driven by a direct-connected Westinghouse one-eighth horsepower motor. A rheostat in the armature of the latter provided means of varying the speed, and up to the limit of its capacity this outfit was the most satisfactory source of air we have ever used, but a somewhat larger capacity was later found desirable. An Eimer and Amend No. 780 blower has proven very satisfactory for this purpose. Small pressure variations were still observed, even after the blower had been installed, and these were found to depend largely on the state of the cement in the separating chamber. It was felt at the time that these pressure variations were unavoidable in this type of separator, which now very closely resembled Petersen's apparatus, as described in the foregoing review.

The third objection to the apparatus—that is, the tendency of the cement to choke and lodge in the cone at the bottom of the separating chamber—proved to be the most difficult of all to overcome, and led eventually to the development of the present form

of analyzer. Numerous attempts were made to avoid this difficulty, one being worthy of special mention. An apparatus was constructed according to the plan shown in Fig. 11. C_1 and C_2 were two brass cones of the same dimensions surmounted by tubes T_1 and T_2, the former connected with the top of the latter by means of a slightly inclined conical tube. T_2 was surmounted by a section of a glass funnel C_3 in the top of which was mounted a cylindrical brass tube T_3, approximately 3 inches in diameter and 3 feet in height. The latter constituted the separating chamber of the apparatus and carried a dust collector on its upper end. The cement to be tested was placed in cone C_1, which was removable. The tube T_1 was partly of glass, to permit observation of the behavior of the cement during the process of blowing. Short lengths of rubber tubing connected to the air supply were attached to the bottom of C_1 and C_2 and closed with pinch cocks to prevent the cement from getting back into the tubes when the air was not flowing. With everything in readiness for the operation, air was first admitted to the cone C_2 and then to the cone C_1. If the air supply were sufficient to handle the cement, the latter was thrown up into the glass tube in violent agitation, and was soon freed from its finest particles. The latter passed over into the tube T_2 and were then further separated, part passing up into the collector, part remaining in suspension in the vicinity of C_3, and the coarsest particles falling back into cone C_2. There was thus a gradual diminution of material in cone C_1, and a gradual accumulation in cone C_2. The obvious purpose of this device was to equalize the retardation of the air stream, thus assuring a more constant flow in the separating chamber, and by using higher pressure with small inlets C_1 and C_2, to induce a more vigorous action in the cement in C_1. Many excellent analyses were made with this device, and photomicrographs of particles showing the sizes and purity of the separations are shown in Figs. 12 to 15. Fig. 16 shows the relative sizes of the largest particles passing a No. 200 sieve.

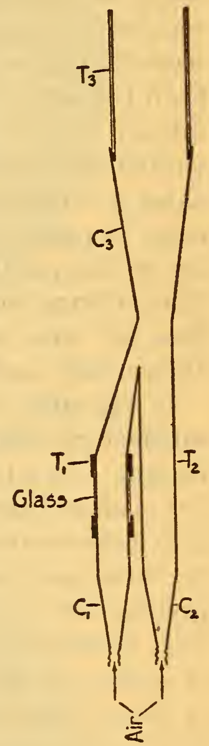

FIG. 11.—*Bureau of Standards double-cone analyzer (discarded)*

Our confidence in this apparatus gradually dwindled, however, when we became assured that it was barely capable of separating less than 30 per cent of a normal cement as the finest fraction obtainable. The size of this separation was in the vicinity of 0.001 inch (0.025 mm.)—that is, the average diameter of the largest particles as seen in the field of a microscope—and the amount of air required to produce this separation was so diminished as to be incapable of handling very finely ground cements. This trouble seemed to depend entirely on the presence of the very fine material, for if the latter were partially removed or the original sample were diluted with a sufficient amount of coarse grains, the process started much more readily. Of course an analysis could be coaxed along by continued tapping, stirring, and by allowing the air to enter in pulses, but for routine analyses such a performance was out of the question. Later experience showed that this difficulty from sticking and clogging might have been overcome, but at that time we came to the conclusion that the up-blast principle was theoretically and practically the weak point in all our elutriators.

It was quite by accident that we hit upon the very simple and satisfactory scheme which has since been adopted in all our later designs. In attempting to blow a considerable quantity of cement out of an ordinary Mason jar with a small air nozzle it was observed that if the nozzle were held stationary and directed into the cement, the latter was blown away, not violently, but by a gradual erosion process, leaving a large conical hole in the mass of the cement. This immediately suggested a new scheme for an analyzer, viz, of sealing up the air inlet in the bottom of the separating cone of the old apparatus and inserting an overhead nozzle *to blow down into the apex of the cone from above.* This was carried out by having a pear-shaped glass bulb blown, into the side of which a bottle-neck opening was made to admit nozzles of glass tubing of different sizes. The open end at the top was also in the form of a wide-mouthed bottle, and designed to fit air-tight to the lower end of a tall glass stack. The original apparatus of this form is still in operation and may be seen in Fig. 18 at *B*. Since this apparatus was constructed improvements have been made in all its parts, but the principle of blowing the air down into the cement from above has been retained and is the chief distinguishing feature of the new analyzer.

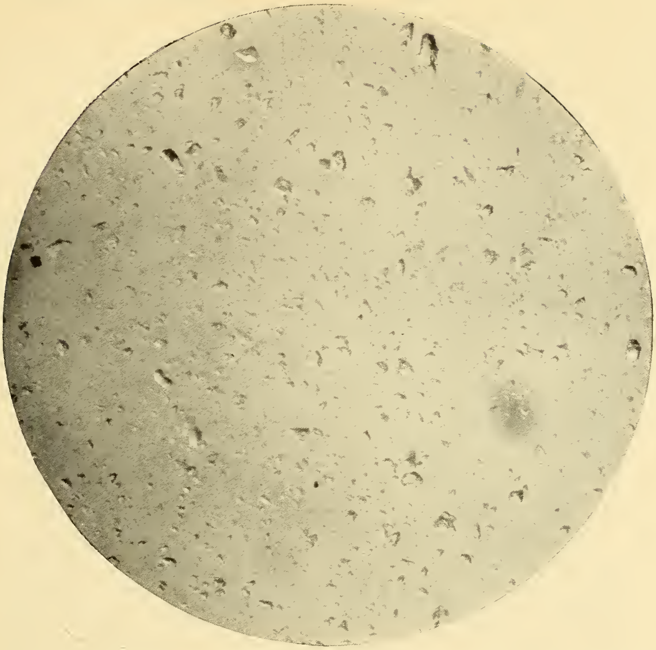

Fig. 12.—*Air-separated cement particles, Grade I, or "flour." Average diameter of maximum particles, 0.0011 inch. Magnification, 150 diameters*

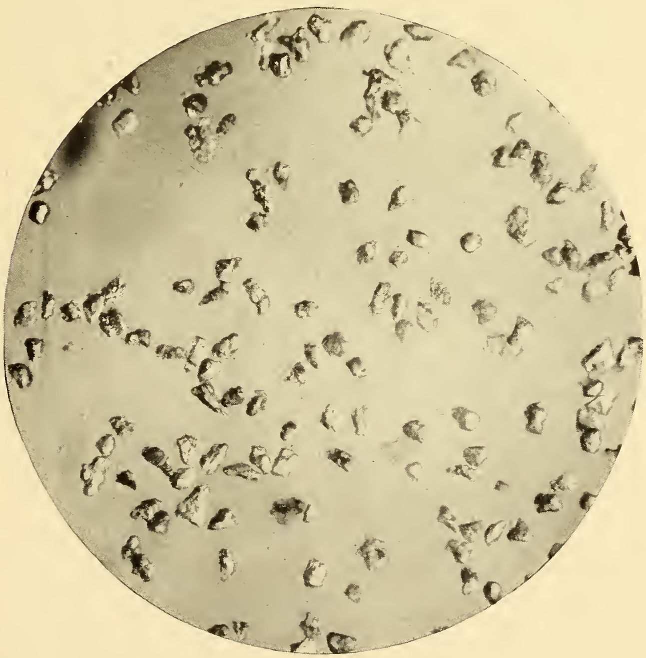

Fig. 13.—*Air-separated cement particles, Grade II. Average diameter of maximum particles, 0.0018 inch. Magnification, 150 diameters*

Fig. 14.—*Air-separated cement particles, Grade III. Average diameter of maximum particles, 0.0025 inch. Magnification, 150 diameters*

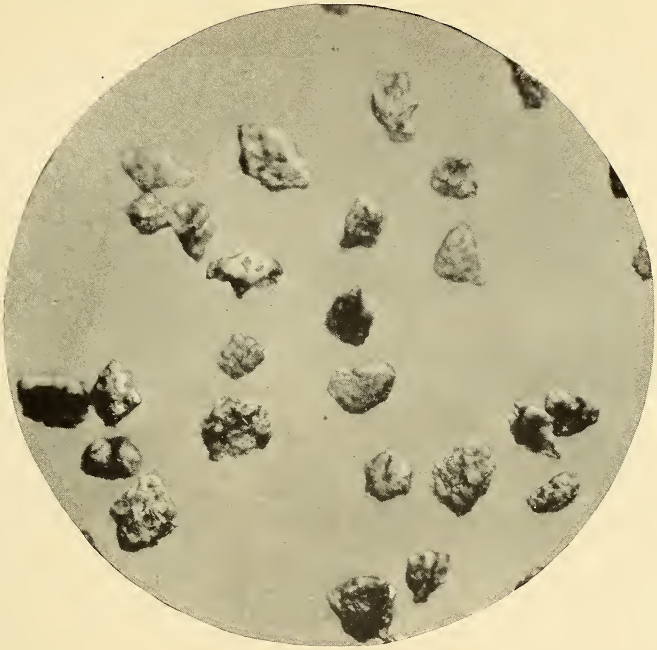

Fig. 15.—*Air-separated cement particles, Grade IV. Average diameter of maximum particles, 0.0033 inch. Magnification, 150 diameters*

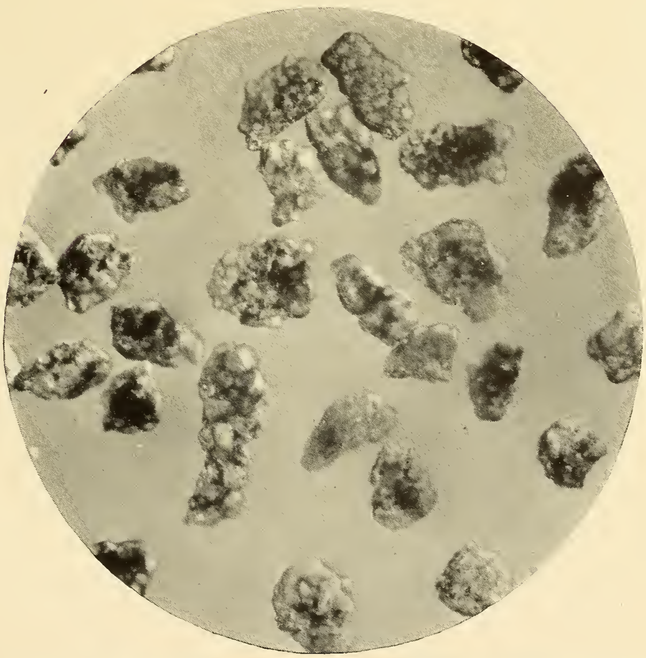

Fig. 16.—*Cement particles passing a No. 200 sieve at the end of the ordinary sieving operation. Magnification, 150 diameters*

V. DESCRIPTION OF THE ANALYZER IN ITS PRESENT FORM

The present analyzer in operating condition is shown in Fig. 17. The analyzer proper is shown at the right of the diagram, and consists of four essential parts—the bulb, with its set of three inter-

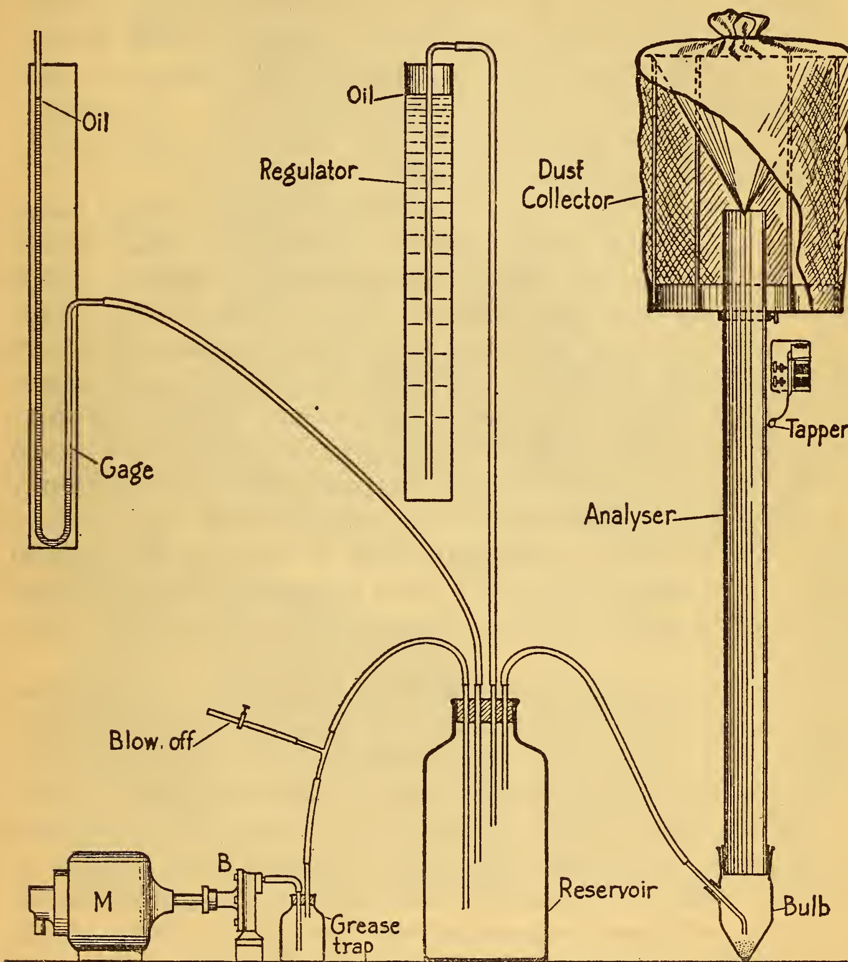

FIG. 17.—*Bureau of Standards air analyzer*

changeable nozzles; the stack, or separating chamber; the tapper, electrically operated; and the dust collector, which retains all material carried off through the separating chamber. The auxiliary apparatus required for the operation of the analyzer consists

of a motor M, direct connected to a blower B, beyond which is a grease trap to prevent oil and grease from getting into the air tube leading to the reservoir. A side valve is inserted in this tube which acts as a blow-off for the large excess of air delivered by the blower over and above the amount passing through the analyzer. The remainder of the air passes into a reservoir to which are connected an oil-pressure gage, a regulator, and finally the tube leading directly to the nozzle in the analyzer bulb. The auxiliary apparatus is designed entirely for the purpose of automatically supplying air at constant pressure to the analyzer.

It has been stated that a satisfactory elutriator should insure as far as possible a constant velocity and uniform stream lines in the fluid as it passes through the separating chamber. In our apparatus the separating chamber is a drawn brass tube 6.8 cm (2.7 inches) internal diameter, 7 cm external diameter, 150 cm (60 inches) long, polished on the inside without a projection of any sort on the inside walls. The air expands immediately after striking into the cement, and aside from the unavoidable pulsations and eddies in the bulb should have every opportunity to flow uniformly up the stack. Experiments will be described later which tend to show that the stack is sufficiently high to insure uniform stream lines and to indicate that the friction between the air stream and the polished surface of the stack has a negligible influence on the separations.

The second requirement of a satisfactory elutriator is that neither the cement nor the dust collector should retard the air delivery, unless such retardation be compensated in some way. This requirement is met particularly well by the distinguishing feature of the apparatus, viz, the delivery of the air into the cement from an overhead nozzle, and by the type of collector which is shown in Fig. 17. That any appreciable back pressure arising from the use of a fair sized flannel sack as dust collector did not exist was shown by attaching a small pressure gage to the top of the glass stack used with the original pear-shaped bulb. This gage can be seen at the top of the analyzer marked B in Fig. 18. Even with the maximum air delivery, this pressure did not amount to more than a millimeter of kerosene. That the air delivery from the nozzle

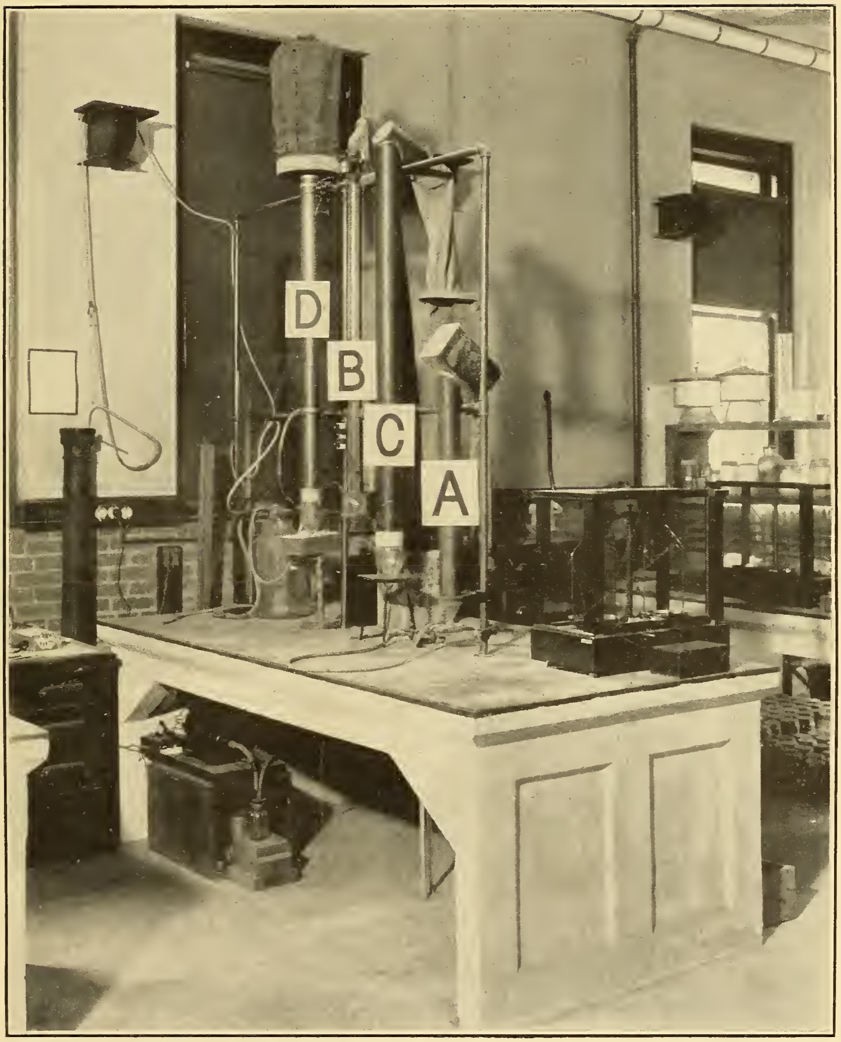

Fig. 18.—*Section of laboratory equipped for air analysis of cements.* A, *an early form of "up-blast" analyzer, now used for removing dust from samples of cement and other materials.* B, *the original apparatus of the "down-blast" type. Very fine separations can not be made with this analyzer owing to small diameter of stack. Calibration is also uncertain because of the difficulty of getting pure slides at the end of an analysis.* C, *improved form of analyzer, better adapted to fine separations on account of its larger stack.* D, *present form of analyzer designed to give three well-distributed separations (four fractions) of the portion of cement passing a No. 200 sieve. The shape of the bulb and the collector of this apparatus are the chief improvements over analyzer* C

just above the cement is inappreciably retarded by the latter was to be inferred from early attempts to observe how much pressure change in the reservoir might be produced by bringing the free-blowing nozzle up toward a flat plate in such way that the air stream from the nozzle impinged vertically on the plate. No appreciable change was observed with maximum air delivery until the nozzle had approached to within 1 cm of the plate. In the present form of apparatus the combined retarding effect of cement and dust collector was determined by metering the air delivery from the separate nozzles, first when blowing free into the atmosphere, and again when blowing into the analyzer under normal operating conditions. With the largest nozzle used, the observed difference in air delivery under the two conditions was less than 1 per cent, an amount which theoretically affects the size of separation by less than 0.5 per cent.

Another requirement of a satisfactory analyzer is that the cement shall be completely and continuously exposed to the action of the air, otherwise complete and clean separations will not be obtained. In this apparatus the action depends entirely upon the air stream from the overhead nozzle having sufficient energy to penetrate to the apex of the bulb. It is obvious, therefore, that the separation of the finest fraction, requiring the use of the smallest nozzle and the minimum air delivery, is the only one which needs careful attention, for if this separation can be made satisfactorily, the coarser separations, requiring greater air delivery, will certainly cause no trouble. The most important variables which may effect the proper penetration of the air stream to the bottom of the cement are (1) the pressure of air in the reservoir, (2) the diameter of the stack, (3) the diameter of the bulb, (4) the shape of the bulb, (5) the diameter of the nozzle, and (6) the quantity of cement. More or less attention has been devoted to all these items in the development of the analyzer, and we have found that the most feasible method of operation is to choose a stack of such diameter that three different separations—that is, four fractions—can be readily obtained in the cement passing a No. 200 sieve.

It will be shown later that the stack used in the present form of apparatus, 6.8 cm (2.7 inches) internal diameter, is probably quite near the most efficient size to use for these three separations.

The diameter of the stack practically determines the diameter of the bulb, for the latter is at best very slightly larger than the diameter of the stack, thus allowing the attachment of the bulb to the gently tapered collar at the bottom of the stack. The interior of the top of the bulb is ground to the same taper as the brass collar, but slightly larger, so that it may fit closely over a thin strip of soft leather glued to the outer surface of the collar. This provides a perfectly dust-tight connection between bulb and stack, at the same time reducing danger of breakage and facilitating removal of the bulb for weighing. The extreme height of the bulb as represented in Fig. 17 is 19 cm (7.5 inches), the apex being 14 cm (5.5 inches) below the bottom of the stack. It has also been found most feasible to operate the analyzer with a constant air pressure in the reservoir, which is automatically kept at 1 pound per square inch (0.07 kg per square centimeter) in our present series of experiments.

Our aim has been to procure the various separations desired by simply changing nozzles, a set of which are ground with the same taper and fit into the bulb in the manner shown in Fig. 17. These nozzles are all adjusted to point vertically downward into the apex of the bulb. The original tentative selection of nozzles was based on the sizes of separation desired, and as the average diameter of the largest particles passing the No. 200 sieve is approximately 0.004 inch (0.1 mm), it was suggested that 0.003 inch (0.075 mm), 0.002 inch (0.05 mm), and 0.001 inch (0.025 mm), would be desirable limiting sizes for the finer separations. While these are not the exact sizes of the separations now obtained, and, in fact, differ considerably from what may be regarded as the true sizes of separation, we are accustomed to refer to the three nozzles as the 0.001, the 0.002, and the 0.003 inch nozzles, respectively, and for convenience they will be thus designated hereafter, although it should be always remembered that these are not the true sizes.

Referring to the statement at the beginning of the second paragraph, on page 27, it is the 0.001-inch nozzle which alone may not deliver air with sufficient energy to insure the complete stirring and mixing of the residue in the bulb. It happens that the

0.001-inch nozzle in the present apparatus delivering air at 1 pound per square inch is just capable of handling a 50 g sample of cement, but this was not accomplished without a number of attempts to improve the form of the conical part of the bulb. The latter is now a true cone with a slightly rounded apex, and an angle of 63°. The depth of the cement may be reduced by using a smaller sample, or by flattening the cone of the bulb, and a cone of 70° would probably give even better results than the one now in use. In routine examinations we are accustomed to use 33⅓ g of cement for the 0.001-inch analysis, which gives practically the same results as the 50 g sample and in less time. With a given size of stack and a fixed definite pressure in the reservoir, the diameters of the nozzles are practically predetermined. As the analyzer is calibrated entirely from the separations, however, we have never measured the nozzles accurately, and it may be emphasized here that neither the sizes of the nozzles nor any other dimensions of the apparatus, nor even the working pressure, need to be specified or known with accuracy. As a matter of record, the internal diameters of the nozzles now in use are approximately 1.1 mm (0.04 inch), 2.2 mm (0.09 inch), and 3.3 mm (0.13 inch), respectively.

The fourth important requirement of a good analyzer is that it should have no places of lodgment for material in the separating chamber. This is an obvious requirement for sharp and complete separations, and means that whenever an analysis is interrupted for any reason, all the material should soon settle either in the dust collector or in the residue in the bottom of the analyzer. All possible precautions have been taken to insure complete separations in this apparatus, and the following features contribute especially to this end: (1) The stack is polished on the interior surface, but in spite of this a small amount of the finest dust will gradually collect in the upper part of the stack, and in a single analysis may amount to a few hundredths of a gram; (2) the upper rim of the stack is filed to a sharp knife-edge beveled outward and downward, so that any particles which might otherwise lodge on the rim are deposited in the collector; (3) an electrical tapper is mounted near the top of the stack, and is ordi-

narily kept running at the beginning of an analysis to assist in circulating the cement in the bulb, and at the end of an analysis to prevent all possible adherence of particles to the inside of the stack when the rates of loss are being determined; (4) the dust collector has been designed especially to prevent any material from falling back into the residue when it has once made the trip through the separating chamber.

The essential feature of the dust collector is a 60° cone of polished sheet copper, supported by eight rods soldered at regular intervals to the inside of the rim of the brass pan at the bottom, and so arranged that the apex of the cone projects slightly into the upper end of the stack. The effect of this device is to moderately accelerate the air stream as it leaves the stack without causing any appreciable retardation in the uniform air flow up the main part of the stack. At the same time the air stream is diverted symmetrically outward into the body of the collector without diminishing the vertical component of flow until the particles in suspension are safely carried beyond the region directly above the mouth of the stack. The frame of the collector carries a loose covering of canton flannel taped tightly around the pan and gathered at the top, thus providing an efficient filter for the more or less dust-laden air stream without causing an appreciable back pressure. In the center of the pan is fastened a brass sleeve which fits closely around the stack and insures the rigidity of the collector, and the whole is supported in its proper position by an adjustable clamp. The frame of the collector is approximately 25 cm (10 inches) in diameter and 50 cm (20 inches) high.

VI. METHOD OF OPERATION—SPECIMEN ANALYSES

The complete process of making a separation with the analyzer is carried out as follows:

The motor and blower are first started at slow speed, and the air tube leading from the reservoir to the analyzer is connected to the nozzle to be used. This causes a rise in pressure in the reservoir, indicated on the gage, which is to be raised to the work-

ing pressure of 1 pound per square inch. The pressure is further raised by gradually closing the blow-off, and if not high enough when the latter is completely closed, the blower speed is increased. It is desirable always to have an excess of air supplied by the blower, and to have the blow-off so adjusted as to allow a slightly greater quantity of air to pass into the reservoir than is required for the analyzer. Further regulation of pressure is automatically provided by the regulator, which consists of a vertical pipe about 5 feet (150 cm) long and 4 inches (10 cm) in diameter, closed at the bottom and nearly filled with kerosene. Into this a long glass tube connected to the reservoir and open at the lower end projects to a depth which can be adjusted and is approximately the same as the difference in level of the kerosene in the two arms of the gage at working pressure. This adjustment is always made by trial, and when the proper depth is attained, the regulator functions perfectly for an indefinite time without further attention.

It is obvious that if the pressure in the reservoir is below the required working pressure, air can escape from the reservoir only through the analyzer nozzle, but by further closing the blow-off the pressure rises and the kerosene is driven down the regulator tube until finally the seal is broken and air bubbles off. The pressure in the reservoir will thereafter remain sensibly constant, unless the speed of the blower varies considerably. In normal operation, therefore, the oil in the pressure gage mounts quickly to its prescribed height and remains there while the regulator disposes of the slight excess of air supplied to the reservoir. Unavoidable irregularities in the speed of the blower are thus automatically compensated and the gage indicates the constant pressure of the air supplied to the nozzle. Variations in the reservoir pressure as large as 1 per cent are rare, and of this magnitude are entirely negligible in their effect on the separations.

Having adjusted the blow-off, as previously described, the nozzle is removed from the air tube and inserted in the bulb, which is detached from the stack. The weight of nozzle and bulb should be known to the nearest 0.01 g. If the 0.001-inch separation is to be made, a 33⅓ g sample of cement is placed in the bulb; if the coarser separations are desired, 50 g are ordina-

rily used. The bulb containing the cement is then attached to the stack, the air tube is connected to the nozzle, the tapper is started, and the analysis proceeds without further attention on the part of the operator. The residue in the bulb gradually darkens as the fine material is removed and in the course of half an hour or less appears to become distinctly granular, especially in the coarser separations. It has been found by experiment that greater uniformity in the fractions is obtained if the separations are regarded as completed when a certain rate of loss is reached, as in the case of the No. 200 sieve fineness determination. The air separations require a considerably longer time, however, as the diminution of the quantity of material removed is much less rapid toward the end of the process than in the sieve separations. The rate of progress of the air analyses is shown in Fig. 19.

We have arbitrarily adopted a loss of 0.02 g per minute as the most suitable rate to indicate the completion of a separation, but should the analyzer be found adapted to routine work, a considerable saving of time might be effected without much sacrifice of accuracy by adopting a higher rate. The rate of loss is determined in the following manner: The approximate time of an analysis is usually known from experience, and 15 or 20 minutes before the estimated time of completion the air tube is detached from the nozzle, and a half minute or more, depending upon the size of separation, is allowed for the particles suspended in the stack to settle back into the bulb. The latter is then removed and weighed. It is then replaced on the stack and the analysis is continued for exactly 10 minutes, after which the bulb and residue are again weighed. If the observed loss is greater than 0.2 g, the analysis is continued for another 10 minutes, and so on until a loss of 0.2 g or less has been observed in the 10-minute interval. The analysis is then completed, and the total loss computed to the minute when approximately 0.02 g was being removed. The total loss is expressed in percentage of the total cement, and check tests usually show an agreement within 0.5 per cent.

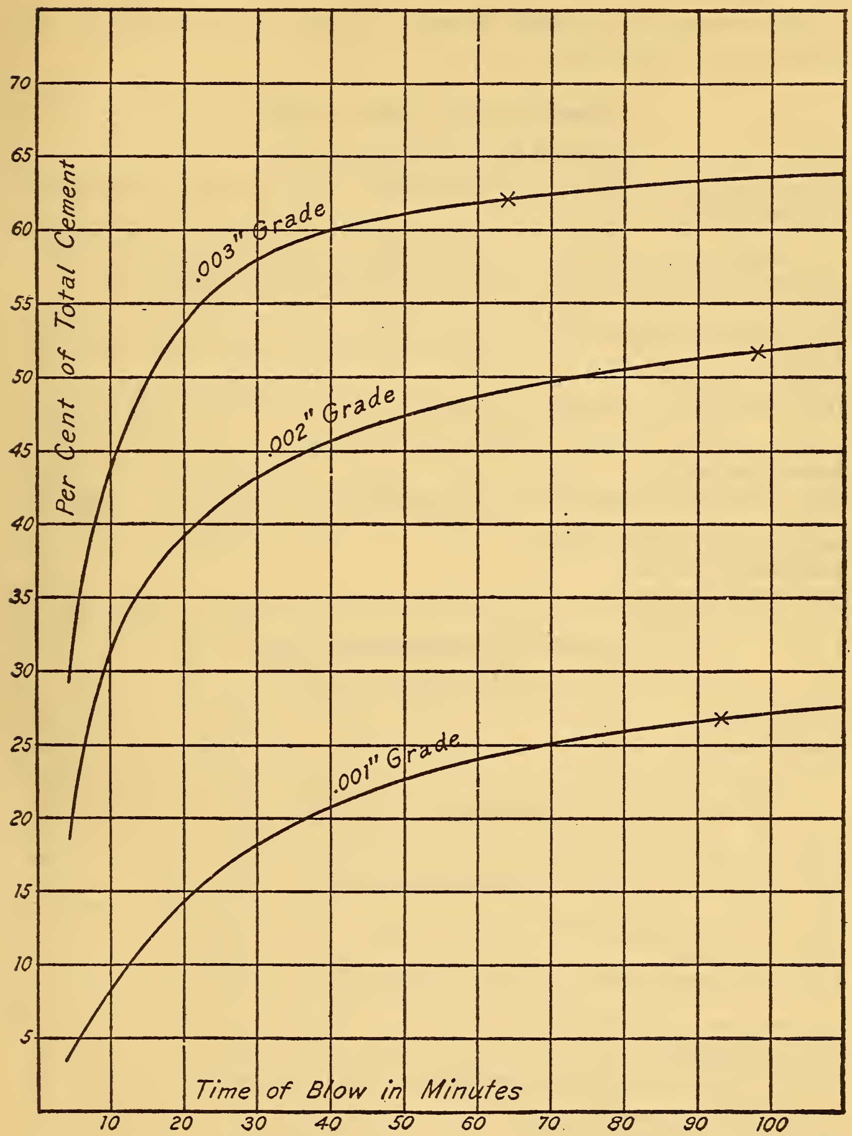

FIG. 19.—*Curves showing rate of removal of fine material in air analyses; 33⅓-gram samples used. The cement from which these curves were obtained is peculiar in having an unusually low "flour" content (0.001-inch grade) in comparison with the quantities of the coarser grades, 81 per cent passing the No. 200 sieve. The end points of the analyses are indicated by crosses*

Three specimen analyses, exactly as made and entered in our records, are given below:

Cement No. 406,[a] 0.001-inch nozzle

[Monday, Dec. 7, 1914, began at 4.17 p. m.]

Weight of bulb	251. 10 g
Weight of cement	33. 33
Total	284. 43
(Analysis interrupted at 4.43 p. m. and continued Dec. 8, beginning at 8.58 a. m.)	
Weight of bulb and residue after 80 minutes blow	274. 67
Loss in 80 minutes	9. 76
Weight of bulb and residue after 90 minutes blow	274. 44
Loss from 80th to 90th minute	0. 23
Loss in 90 minutes	9. 99
Weight of bulb and residue after 100 minutes blow	274. 27
Loss from 90th to 100th minute	0. 17
Rate of loss in 90th min	0. 02
Total loss in 90 minutes	9. 99 g
	= 29. 97%

Cement No. 406, 0.002-inch nozzle

[Dec. 8, 1914, began 11.22 a. m.]

Weight of bulb	251. 10
Weight of cement	50. 00
Total	301. 10
Weight of bulb and residue after 90 minutes blow	276. 44
Loss in 90 minutes	24. 66
Weight of bulb and residue after 100 minutes blow	276. 18
Loss from 90th to 100th minute	0. 26
Loss in 100 minutes	24. 92
Weight of bulb and residue after 110 minutes blow	275. 95
Loss from 100th to 110th minute	0. 23
Loss in 110 minutes	25. 15
Weight of bulb and residue after 120 minutes blow	275. 77
Loss from 110th to 120th minute	0. 18
Rate of loss in 111th minute	0. 02
Total loss in 111 minutes	25. 17
	= 50. 34%

[a] Cement No. 406 is one of the standard samples furnished by the Bureau for tests of No. 200 sieves.

Cement No. 406, 0.003-inch nozzle

[Dec. 9, 1914, began 10.30 a. m.]

Weight of bulb	251. 55 g
Weight of cement	50. 00
Total	301. 55
Weight of bulb and residue after 70 minutes blow	272. 83
Loss in 70 minutes	28. 72
Weight of bulb and residue after 80 minutes blow	272. 60
Loss from 70th to 80th minute	0. 23
Loss in 80 minutes	28. 95
Weight of bulb and residue after 90 minutes blow	272. 40
Loss from 80th to 90th minute	0. 20
Loss in 90 minutes	29. 15
Weight of bulb and residue after 100 minutes blow	272. 23
Loss from 90th to 100th minute	0. 17
Rate of loss in 85th minute	0. 02
Total loss in 85 minutes	29. 05
	= 58. 10%

The actual records are of course in more condensed form, each step in the above analyses being fully indicated for the sake of clearness. The operator also carefully notes the time of beginning each 10-minute check test. The stopping point is determined by simple linear interpolation of rates and is approximate only, but quite close enough for all practical purposes. This is carried out as follows for the 0.003-inch separation: 0.023 g is taken as the rate of loss at the seventy-fifth minute, 0.020 g as the rate at the eighty-fifth minute, and 0.017 g at the ninety-fifth minute. The eighty-fifth minute is therefore taken as the stopping point. The loss in 80 minutes was 28.95 g, and the rate of loss at the eightieth minute was assumed to be the mean rate between the seventy-fifth and eighty-fifth minutes, or 0.0215 g. Similarly the mean rate from the eightieth to the eighty-fifth minute is 0.0208 g and the loss from the eightieth to the eighty-fifth minute is 5×0.0208, or 0.10 g. Hence the total loss in 85 minutes is 29.05 g. While this procedure illustrates every step of the process, a simple inspection is generally all that is necessary to determine both the stopping point and the total loss.

The foregoing separations, combined with the fineness as determined on the No. 100 and No. 200 sieves, give a division of the

cement into six fractions. These fractions can be compared with similar fractions of other cements obtained in the same way, and thus interesting information can be obtained regarding the performance of different finishing mills, the increase in the finer grades by regrinding, and the relation between the amount of the 0.001-inch grade, which may be provisionally called "flour," and the amount passing the No. 200 sieve, which is the present method of judging the fineness of different cements.

VII. CALIBRATION OF THE ANALYZER—METHODS OF MEASURING PARTICLES

The quantitative separation of cement into fractions, as described in the preceding section, constitutes an analysis which can be interpreted only with reference to the particular apparatus employed and is not an absolute mechanical analysis. The very great desirability of obtaining comparable analyses of a given cement from different analyzers is in itself sufficient justification for earnest efforts to interpret the separations in terms of size of particles, and if this can be accomplished with even a fair degree of success it carries with it many practical advantages. For example, if we are assured that an analyzer is correct in principle both as to construction and operation, the determination of its sizes of separation does away with the necessity of knowing or referring in any way to its dimensions or to the details of operation; and the laborious adjustment of many parts, which must otherwise be carefully constructed and tested for the purpose of obtaining reliable comparisons, can be entirely avoided. On the other hand, the microscopic study of particles shows that the sizes of separation are not entirely dependent on the analyzer but depend also upon the peculiarities of the cements and possibly other conditions. Thus our problem is to determine first the nominal sizes of separation and then to establish in so far as possible the influence of various factors or conditions on the separations. The study of the latter part of the problem is a long investigation in itself and is logically preceded by a discussion of the methods of measuring particles and a selection of some working method which may be either regarded as a standard system of measurement or reducible to a standard system. The method of

calibrating the analyzer which we have provisionally adopted is entirely covered in the following discussion of methods of measuring particles.

Ambiguity arises from the fact that cement particles are not spheres but irregularly shaped fragments resembling lumps of coal or unground cement clinker. The true sizes—that is, the volumes—of particles can therefore only be approximately determined, and the most feasible method appears to be the averaging of a large number of microscopic measurements of individual particles. As a rule the length, breadth, and thickness of particles as seen on a microscopic slide are fairly definite dimensions which can be measured, and we have to assume, as in the case of larger particles, that the best approximation to volume is obtained by regarding the particles as ellipsoids whose axes are these dimensions. We then have as the volume of the particle

$$V=\frac{\pi}{6}L\times B\times T$$

where L, B, and T are the observed length, breadth, and thickness. The volume of an equivalent sphere is given by the expression

$$V=\frac{\pi}{6}D^3$$

where D is the diameter; and from these two relations

$$D=\sqrt[3]{L\times B\times T}$$

in which D is taken as the true mean diameter of the particle. But the measurement of three dimensions of fine particles is too laborious for routine calibrations, and it is desirable to find out whether some simpler system of measurement can not be adopted from which the true sizes may be deduced with considerable accuracy.

In our study of this phase of the problem measurements have been made on several thousand particles by different methods, but not all the sets are directly comparable and additional work needs to be done. Before making such comparisons as are possible the method of taking the slides should be described. When a separation has been completed, as described on page 32, the collector sack is opened at the top and pushed down over the

frame, exposing the open end of the stack. An elliptical piece of cardboard is then inserted obliquely into the top of the stack in such manner as to deflect the air stream almost entirely to one side of the copper cone. The bulb and residue are then replaced and the blow continued for several minutes. In order to obtain slides of the desired purity it is essential that all dust be first allowed to settle in the vicinity of the collector and that the analyzer be secure from the slightest jar. The slide is then held horizontally just outside and below the rim of the stack on the side to which the air stream is deflected by the card. From one to three minutes are usually required to obtain a slide having the desired number of particles.

The method of measurement originally adopted by us, and the method by which most of our measurements have been taken, was one which first seemed capable of giving the greatest uniformity, although it does not represent even approximately the true size of separation. In this method the diameters of the largest particles are measured—that is, the particles which appear to be largest—irrespective of their orientation. In all our measurements a Bausch and Lomb petrographic microscope, with filar micrometer eyepiece and mechanical stage attachment, has been used. In measuring the particle diameters, which are always taken in the direction of travel of the movable cross hair, the latter is brought tangent first to one edge and then to the other, changing the focus when necessary to obtain sharp settings. The difference between the readings of the micrometer head in the two positions, multiplied by the value of the divisions in inches or millimeters, is taken as the diameter of the particle.

This system of measurement was followed for more than two years, for we believed that it not only afforded the greatest uniformity but also indicated the extreme limiting sizes of any given separation. We have recently discarded this system, however, after a series of comparisons with other methods, in favor of an average measurement of all the particles on a given slide. In the latter system an observer starts, let us say, at one corner of a slide, noting the readings of the two verniers on the mechanical stage. He measures the particle which is intersected by the fixed cross hair nearest the center of the field and then moves across

the slide measuring particles every 2 or 3 mm in a similar manner. The slide is then moved along a certain distance, depending upon the total number of measurements desired, and another line of particles across the slide is measured. In this manner the entire slide is covered and an average diameter of the particles is obtained from a purely mechanical selection.

TABLE 1

Uniformity of Results in Measuring Maximum Particles and Average Particles

Cement	Grade	Diameter of maximum particles by 3 observers				Diameter of average particles by 3 observers			
		P	S	G	Range	P	S	G	Range
		Inch	Inch	Inch	Inch	Inch	Inch	Inch	Inch
406........	100 mesh..	0.00929	0.00893	0.00860	0.00069	0.00786	0.00812	0.00781	0.00031
406........	200 mesh..	.00528	.00510	.00485	.00043	.00414	.00439	.00430	.00025
3008.......	0.003 inch.	.00286	.00296	.00307	.00021	.00221	.00235	.00227	.00014
3296.......	...do......	282	280	268	14	201	210	193	17
3296.......	...do......	292	280	289	12	208	234	213	26
3457.......	...do......	280	264	270	16	209	216	199	17
3457.......	...do......	269	289	298	29	216	215	213	3
3693.......	...do......	309	287	291	22	222	222	224	2
3693.......	...do......	303	284	304	20	216	221	223	7
406........	...do......	270	268	277	9	212	203	211	9
406........	...do......	268	268	276	8	217	226	225	9
Mean..	....	.00284	.00280	.00287	.00017	.00214	.00220	.00214	.00012
3008.......	0.002 inch.	.00203	.00199	.00206	.00007	.00150	.00139	.00148	.00011
3008.......	...do......	207	203	213	10	150	166	159	16
3296.......	...do......	204	204	199	5	143	164	145	21
3296.......	...do......	202	202	199	3	155	168	158	13
3457.......	...do......	188	192	198	10	143	147	141	6
3457.......	...do......	196	200	209	13	150	161	156	11
3693.......	...do......	210	218	200	18	165	164	157	8
3693.......	...do......	214	207	220	13	168	163	161	7
406........	...do......	190	182	187	8	142	152	151	10
406........	...do......	184	180	183	4	150	152	148	4
Mean..	....	.00200	.00199	.00201	.00009	.00152	.00158	.00152	.00011
3008.......	0.001 inch.	.00098	.00112	.00102	.00014	.00079	.00078	.00076	.00003
3296.......	...do......	101	106	98	8	74	80	73	7
3457.......	...do......	93	103	91	12	74	70	68	6
3457.......	...do......	96	92	98	6	77	74	76	3
3693.......	...do......	104	100	98	6	74	75	79	5
3693.......	...do......	105	107	109	4	76	78	80	4
406........	...do......	102	98	98	4	76	81	77	5
406........	...do......	94	98	100	6	72	80	72	8
Mean..	....	.00099	.00102	.00099	.00008	.00075	.00077	.00075	.00005

Table 1 contains all comparable sets of measurements made by the two methods described in the foregoing paragraphs. In general, each line of the table contains measurements by three observers on a single slide, but in a few cases on two similar slides. Columns 1 and 2 contain the laboratory numbers and the nominal sizes of separation of the cements used; columns 3, 4, 5, 7, 8, and 9 contain the individual measurements by three observers, each recorded value being the average diameter of 40 particles; and columns 6 and 10 show the ranges of the three observers' determinations in the two methods of measurement.

It is to be noted that from the average ranges for each grade that in all cases except one the selection of average particles gives more uniform results than the selection of the largest particles. It was anticipated also that in the selection of the largest particles personal differences would be more apt to appear than in the mechanical selection of average particles. In our earlier measurements this was undoubtedly the case, but the three observers responsible for the measurements herein reported show little variation in the selection of maximum particles, whereas observer S shows slightly higher values throughout in the selection of average particles. The only explanation that may be offered to account for this discrepancy is that the observer had had no previous experience in microscopic measurements and in the earlier part of the work had not understood that the focus should be changed, if necessary, in the two settings on each particle. This probably resulted in slightly larger diameters, and may account for the peculiarity noted. Certainly only very slight personal differences should be looked for in the selection of average particles, as indicated by the agreement of the average measurements of observers P and G.

There are also further objections to the selection of maximum particles for measurement, one of the most obvious being the failure of this system to indicate the true size of separation. It has also been observed, as might be anticipated, that larger diameters are obtained from slides thickly covered with particles than from thin slides, and low magnifications tend to yield higher results than high magnifications, because of the larger field of the former.

But the main objection to this system has been brought out by three dimensional measurements, which establish quite conclusively that the majority of the apparently largest particles as seen in a plane are usually lamellar in form and do not vary so greatly from the true average size as their appearance would indicate. This is shown by the comparison of results presented in Tables 3 and 4. On the other hand, the selection of particles at random—that is, the average selection as described on page 38 is made without the mental effort required in the other system. It represents more nearly the actual size of separation, and can be reduced to the true size if desired with a fair degree of approximation. It does require, however, representative slides of great purity, in the making of which every precaution must be taken to avoid contamination.

Table 2 is prepared from the same data as Table 1, but contains, in place of the individual observers' measurements, the averages of all. Each recorded diameter is therefore an average of measurements on 120 particles. The last column shows the relation between the diameters of particles of the same grades as determined by the two systems of measurement, an average value of the ratio, average diameter to maximum diameter, being derived for each grade. These average values for the 0.001-inch, 0.002-inch, and 0.003-inch grades—that is, for the air separated grades—are in very good agreement, and we may assume the value 0.765 as a very approximate reduction factor from the older system of measurement to the present system. As previously stated, the sieve separations are more strictly on a basis of absolute size than the air separations, consequently less difference between maximum and average sizes are to be expected, and the reduction factors are therefore higher. Furthermore, the No. 200 sieves are relatively much less uniform than the No. 100 sieves, and the reduction factor for the 100-mesh grade is therefore higher than that for the 200-mesh grade.

The three dimensional measurements have thus far been made by one observer, but it is highly desirable that the accuracy and uniformity of such measurements be checked by others. We hope that this phase of the investigation may be further extended, for

the information to be obtained from these studies has a very important bearing on the theory of elutriation as well as on the

TABLE 2

Relation Between the Diameters of Maximum Particles and Average Particles

Cement	Grade	Diameters of maximum particles	Diameters of average particles	Ratio, average diameter to maximum diameter
		Inch	Inch	
406	100 mesh	0.00894	0.00793	0.887
406	200 mesh	.00508	.00428	.842
3008	0.003 inch	.00296	.00228	.771
3296	do	277	201	.726
3296	do	287	218	.759
3457	do	271	208	.768
3457	do	285	215	.755
3693	do	296	223	.754
3693	do	297	220	.741
406	do	272	209	.769
406	do	271	223	.824
Mean		.00283	.00216	.763
3008	0.002 inch	.00203	.00146	.720
3008	do	208	158	.760
3296	do	202	151	.748
3296	do	201	160	.796
3457	do	193	144	.747
3457	do	202	156	.773
3693	do	209	162	.776
3693	do	214	164	.767
406	do	186	148	.796
406	do	182	150	.825
Mean		.00200	.00154	.771
3008	0.001 inch	.00104	.00078	.750
3296	do	102	76	.745
3457	do	96	71	.740
3457	do	95	76	.800
3693	do	101	76	.752
3693	do	107	78	.729
406	do	99	78	.788
406	do	97	75	.773
Mean		.00100	.00076	.760

practical limits of the air analyzer. The measurement of the length, breadth, and thickness of a particle is now made as follows: For the length and breadth measurements the ordinary illumina-

tion from below the slide is used. Our mechanical stage does not revolve, therefore in order to bring the horizontal wire of the micrometer eyepiece parallel to the length of the particle the eyepiece is turned and the length is then measured in the usual manner. The eyepiece is then turned through 90°, approximately, and the tangent positions of the cross hair determine the breadth. In order to measure the thickness a strong side illumination is required, this being provided by an ordinary 40-watt tungsten lamp and a lens which concentrates the light on the particles directly beneath the microscope objective.

For best results the lamp is placed slightly above the level of the objective, in order that the highest points of the particles may be strongly illuminated. The microscope is first focused as closely as possible on the top surface of the glass slide and then upon the highest point of the particle, the difference in the readings of the micrometer head of the focusing screw in the two positions giving the thickness of the particle. This measurement of thickness is of course subject to relatively large error, especially on the very fine particles. Assuming the pitch of the focusing screw to be 0.25 mm (0.01 inch), the thickness of the 0.001-inch particles is such as to require only about one-twentieth of a turn of the screw to pass from the bottom to the top of the average particle. Nevertheless, if the observations are free from systematic error, the mean of a large number should give a fairly dependable result, the approximate accuracy of which can generally be checked by indirect methods. In these measurements we are practically limited to the use of an 8 mm objective, for the working distance of our 4 mm objective is too small to allow the particles to be illuminated from the side, and the 16 mm objective has been found to give too high values, owing to the relatively slow change in focus. A high-power objective with long working distance would therefore improve the thickness determinations, especially of the finer grades.

Tables 3 and 4 contain practically all results of observations that have been made to determine the length, breadth, and thickness of particles. The tables are arranged in the same manner as Tables 1 and 2, each line representing, except in a few cases, the average length, breadth, and thickness of 50 particles of a single

slide. It should be stated that the values in the sixth columns, $\sqrt[3]{L \times B \times T}$—that is, the true diameters—have been computed once for all from the mean values of L, B, and T, and are not the means of $\sqrt[3]{L \times B \times T}$ for each particle separately. This short cut has eliminated a vast amount of computation, and the

TABLE 3

Summary of Three Dimensional Measurements on Maximum Particles

Cement	Grade	L	B	T	$\sqrt[3]{L\times B\times T}$	Objective	Remarks
		Inch	Inch	Inch	Inch		
406	100 mesh	0.00946	0.00770	0.00534	0.00730	16	Sieve 631.
406	do	983	769	534	739	16	Do.
Mean		.00964	.00770	.00534	.00734		
406	200 mesh	.00534	.00425	.00235	.00376	16	Sieve 576.
406	do	527	416	236	373	16	Do.
421	do	564	440	224	381	16	Do.
421	do	552	445	231	384	16	Do.
Mean		.00544	.00432	.00232	.00378		
406	0.003 inch	.00309	.00251	.00107	.00202	16	7-foot stack.
406	do	303	252	102	198	16	Do.
406	do	292	240	94	187	16	6-foot stack.
406	do	303	246	98	194	16	Do.
Mean		.00302	.00247	[a].00100	[a].00195		
406	0.002 inch	.00214	.00163	.00062	.00130	8	8-foot stack.
406	do	201	161	63	127	8	Do.
406	do	200	171	72	135	8	7-foot stack.
406	do	194	165	75	134	8	6-foot stack.
Mean		.00202	.00165	.00068	.00132		
406	0.001 inch	.00107	.00084	.00039	.00071	8	7-foot stack.
406	do	103	82	34	66	8	Do.
406	do	107	84	36	69	8	6-foot stack.
406	do	107	84	34	67	8	Do.
Mean		.00106	.00084	.00036	.00068		

[a] These readings are questionable, see text, pp. 44–46.

values given probably differ by a negligible amount from those obtained by the longer method. It will be noted that all observations on air-separated particles made with the 16 mm objective are regarded as questionable, the thickness measurements being undoubtedly too high. This is indicated in Table 4 by the com-

parison of thickness measurements in the 0.003-inch and 0.002-inch grades, where it is observed that the low magnifications give higher values.

TABLE 4

Summary of Three Dimensional Measurements on Average Particles

Cement	Grade	L	B	T	$\sqrt[3]{L \times B \times T}$	Objective	Remarks
		Inch	Inch	Inch	Inch		
406	100 mesh	0.00845	0.00703	0.00553	0.00690	16	Sieve 631.
406	200 mesh	.00466	.00368	.00298	.00371	16	Sieve 576.
406	0.003 inch	.00242	.00176	[a] .00178	[a] .00197	16	5-foot stack.
406	do	231	176	137	177	8	Do.
3457	do	235	173	[a] 191	[a] 198	16	Do.
3457	do	245	183	133	181	8	Do.
3008	do	240	176	137	180	8	Do.
3693	do	233	188	125	176	8	Do.
Mean		.00237	.00181	.00133	.00178		
406	0.002 inch	.00164	.00123	.00108	.00129	8	Do.
3457	do	177	136	[a] 125	[a] 144	16	Do.
3457	do	168	127	90	124	8	Do.
3008	do	172	134	98	131	8	Do.
3693	do	174	135	95	131	8	Do.
Mean		.00170	.00130	.00098	.00129		
406	0.001 inch	.00086	.00063	.00056	.00067	8	Do.
3457	do	82	59	47	61	8	Do.
3008	do	90	64	52	67	8	Do.
3693	do	90	64	59	70	8	Do.
Mean		.00087	.00062	.00054	.00066		

[a] These readings are questionable and not included in means, see text, pp. 44-46.

The most striking result of comparisons of Tables 3 and 4 is brought out by tabulating the average true diameters of the different grades of all cements side by side as follows:

True Diameters ($\sqrt[3]{L \times B \times T}$)

Grade	Maximum particles	Average particles
	Inch	Inch
100 mesh	0.00734	0.00690
200 mesh	.00378	.00371
0.003 inch	.00195	.00178
0.002 inch	.00132	.00129
0.001 inch	.00068	.00066

The true diameters of the maximum particles are seen to differ but little, relatively, from the true diameters of the average particles, except in the 0.003-inch grade, where the maximum diameter appears to be considerably greater. All the measurements on the maximum particles of this grade, however, were made with the 16-mm objective, and the comparison therefore corroborates the more direct evidence from Table 4 that this objective gives too high values when used for thickness determinations. The same objection applies to the use of the 16-mm objective on the sieve grades, but here the relative error is of course smaller. The three dimensional measurements thus establish the fact that the apparent maximum particles are but little larger, on the average, than the average particles, from which we may infer that the apparent maximum particles are lamellar in form. In fact, many of these particles appear under ordinary observation to be comparatively thin, if one may judge of this by their relative translucency.

Hazen has stated [9] that in examinations of sand particles separated by elutriation methods, he found the true average diameter of such particles to be approximately the same as the shorter diameter as seen in a plane in the ordinary microscopic observations. It is interesting to note that this relation holds very closely between the average breadths and true diameters recorded in Table 4. This is more readily seen by the following tabulation:

Grade	Breadth	True diameter
	Inch	Inch
100 mesh	0.00703	0.00690
200 mesh	.00368	.00371
0.003 inch	.00181	.00178
0.002 inch	.00130	.00129
0.001 inch	.00062	.00066

The only discrepancy amounting to more than 1 or 2 per cent is in the 0.001-inch grade, which confirms, to some slight extent,

[9] Some Physical Properties of Sands and Gravels, Allen Hazen; Twenty-fourth Annual Report of Mass. Board of Health, 1892.

the suspicion that the 8-mm objective may be giving too high results on these very small particles, just as the 16-mm objective was giving too high values on the coarser grades. Nevertheless, the generally excellent agreement between these figures suggests the abbreviated method of simply measuring the breadths of average particles, a method which avoids the laborious measurements of thickness and yet gives the approximate true diameters. It is also interesting to note that liquid elutriations of sand particles can apparently be compared with air elutriations of cement particles on the basis of absolute size without reference to the apparatus used for making the determinations.

One other comparison of data from the preceding tables may be of interest. We should anticipate that the methods of measurement described on page 38 would yield apparent diameters which are approximately the mean of the lengths and breadths of the same or similar particles. The following tabulation of the mean values for the different grades enables this comparison to be made more readily:

Grade	Apparent maximum diameters (Table 2)	Mean of length and breadth, maximum particles (Table 3)	Apparent average diameters (Table 2)	Mean of length and breadth, average particles (Table 4)
	Inch	Inch	Inch	Inch
100 mesh	0.00894	0.00867	0.00793	0.00774
200 mesh	.00508	.00488	.00428	.00417
0.003 inch	.00283	.00274	.00216	.00209
0.002 inch	.00200	.00184	.00154	.00150
0.001 inch	.00100	.00095	.00076	.00074

These values show that if the apparent diameters of particles are measured on a microscopic slide in one direction without regard to the orientation of the particles, the results obtained will be very slightly larger than the mean of the length and breadth measured separately. In the case of particles selected at random this difference is of the order of 2 or 3 per cent.

TABLE 5

Relation of the Apparent Diameters and the True Diameters of Average Particles

Cement	Grade	Apparent diameters of average particles	True diameters of average particles	Ratio, true diameter to apparent diameter
		Inch	Inch	
406	100 mesh	0.00793	0.00690	0.870
406	200 mesh	.00428	.00371	.868
3008	0.003 inch	.00228	.00180	.789
3457	do	.00212	.00181	.854
3693	do	.00222	.00176	.794
406	do	.00216	.00177	.820
Mean		.00220	.00178	.814
3008	0.002 inch	.00152	.00131	.862
3457	do	.00150	.00124	.828
3693	do	.00163	.00131	.804
406	do	.00149	.00129	.866
Mean		.00154	.00129	.840
3008	0.001 inch	.00078	.00067	.859
3457	do	.00074	.00061	.825
3693	do	.00077	.00070	.909
406	do	.00076	.00067	.882
Mean		.00076	.00066	.869

Table 5 contains a retabulation of all the comparable data in Tables 2 and 4 relating to the average or random selection of particles for measurement. The last column of the table contains the ratios of the true diameters to the apparent diameters; that is, the reduction factors for the latter system to the former. It will be observed that the mean ratios for the air-separated grades show a fairly regular increase in the factor with diminishing size of particle. To what extent this is a true increase we are unable to state at the present time. It is conceivable that the very fine particles may have less tendency than the coarser particles to lie on a slide in their most stable positions, owing to possible electrical or other attractions, in which case we should expect to find less differences between the apparent and true diameters, and consequently a higher reduction factor for the smaller particles. This may also be shown by tabulating the relative proportions of

the mean values of the three dimensions of the air-separated grades in Table 4. Thus, we have—

Grade	L	B	T	Relative proportions of L, B, and T
	Inch	Inch	Inch	
0.003 inch	0.00237	0.00181	0.00133	1:0.764:0.561
0.002 inch	.00170	.00130	.00098	1:0.766:0.576
0.001 inch	.00087	.00062	.00054	1:0.713:0.621

These proportions are somewhat irregular, but they do indicate an increase in the proportionate thickness of the finer particles. On the other hand, there is undoubtedly a tendency to overestimate the thickness by a more or less definite amount, depending on the magnification, this amount being relatively greater for the smaller particles if the same magnification is used throughout, as it was in these observations. We are inclined to believe, therefore, that while there may be a real increase in the reduction factor for small particles, it is probably not as great as indicated by the results in Table 5, and the maximum value for the particles included in these measurements is probably not over 0.85.

Our experience has indicated that certain modifications of the method of measuring three dimensions of microscopic particles will considerably reduce the labor of such observations and probably give equally good results. For example, if measurements on 50 particles are desired to determine the true average diameter of the particles on a given slide, it will require considerably less time and energy on the part of the observer if he measures the length and breadth on one set of 50 particles and the thickness on another set of 50. This is not wholly a matter of convenience for the observer, for, in general, a lower magnification is better adapted for the length and breadth measurements than for the thickness measurements, which appear to require the highest magnification that can be used. For example, a particle of the 0.002-inch grade requires only a 16 mm objective (total magnification 125) for a sufficiently accurate measurement of length and breadth, but an 8 mm objective (total magnification 260), or preferably a 4 mm objective (total magnification 560), for the thickness determination.

We have also considered the use of a micrometer ocular ruled in 0.1 mm cross section for the length and breadth determinations, but have not yet had an opportunity to test its usefulness. It seems probable that this device, if found adapted to these measurements, will save considerable time in calibration work.

The comparisons given in Tables 2 and 5 show what large discrepancies may arise in arbitrary methods of measuring and expressing sizes of particles. The question to be decided, therefore, is whether it is more desirable to report the mechanical analyses obtained with the air analyzer in terms of the apparent sizes determined by routine measurements, or to report them in absolute sizes determined by three dimensional measurements, trusting in the reliability of a reduction factor to reduce apparent sizes to absolute sizes when the latter are not directly determined. Theoretically, we should deal with absolute sizes; practically it is much more convenient to determine relative sizes by a simple method of measurement, and if the same system be followed in all analyses, there should be no objection to reporting results exactly as obtained. Thus, in ordinary mechanical analyses of sand no attention is paid to absolute sizes of grains but the nominal sizes of the screen openings are taken as the sizes of separation. These nominal openings are always less than the absolute size of separation, partly because some screen openings, especially in the finer sieves, are always larger than the nominal opening, partly because the nominal size corresponds more nearly to the mean of the breadth and thickness of the largest particles than to the mean of length, breadth, and thickness. These two factors have the combined effect of making the nominal screen opening correspond very nearly to the average thickness; that is, the average smallest dimension of the particles representing the size of separation. This is shown by data in Table 4, in which the thickness of the 100-mesh and 200-mesh particles were determined as 0.00553 and 0.00298 inch, respectively, the nominal 100-mesh and 200-mesh openings being 0.0055 and 0.0029 inch, respectively. In the routine measurements of microscopic particles, however, the nominal sizes of separation are very approximately the mean of the length and breadth, and therefore greater than the absolute sizes. In general, these nominal sizes of the air-separated grades

are about as much greater than their true sizes as the true sizes of the screened particles are greater than their nominal sizes, so that if the sieve grades are to be represented together with the air-separated grades by a complete mechanical analysis curve the sizes of the sieve separations must be taken in the same manner as the air-separated grades and not on the customary basis of nominal screen opening.

Chiefly on account of the simplicity of the method of measurement described at the bottom of page 38 we have adopted this system for the present as the basis of our mechanical analysis curves for cement. On this basis the nominal sizes of separation for the present analyzer, as obtained from the mean values in Table 2, are—

Grade	Size of separation (Inch)
100 mesh	0.00793
200 mesh	.00428
0.003 inch	.00216
0.002 inch	.00154
0.001 inch	.00076

the cements from which these values were derived representing a considerable variation in physical characteristics. Comparatively few observations have been made on the sieve separations, and some further measurements should be made to establish these sizes with greater certainty, but the foregoing values are probably quite approximate. It should be noted that the calibrations on the different cements differ appreciably, though not greatly, from each other and that the nominal sizes given above are the mean values obtained from a large number of observations. For practically all normal cements the nominal sizes may be considered the actual sizes of separation; only in special investigations will it be necessary to calibrate the analyzer from measurements on the particular material under observation.

Fig. 20 shows a complete mechanical analysis of the portion of cement No. 406 passing the No. 200 sieve, which may be considered typical of normal cements. In this diagram the mechanical analysis curve is plotted in the usual manner, the abscissae representing the sizes of separation and the ordinates representing the cumulative percentage of total material below the corresponding size of separation. The curve is drawn from the results

obtained with three different analyzers, shown at *B*, *C*, and *D*, Fig. 18. Of these analyzers, *D* must be considered the standard. Analyzer *C* is also capable of good separations and fairly close calibration, but because of its larger stack only the 0.001-inch grades and the 0.002-inch grades were obtained with it. Analyzer *B*, the original glass apparatus, gives fairly good separations, but is not adapted to close calibration. Because of its narrow stack, analyzer *B* gives only the 0.002 inch and 0.003 inch separations.

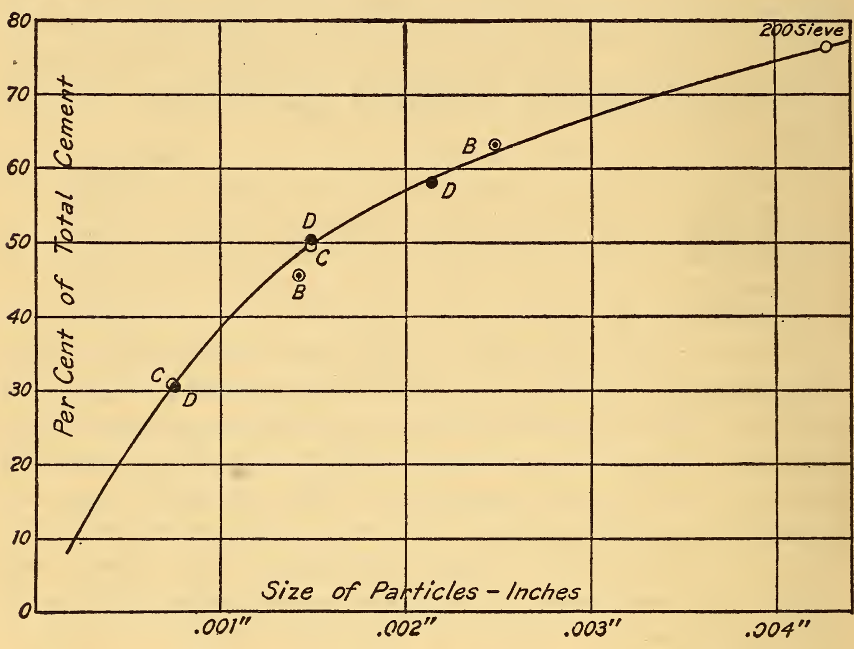

Fig. 20.—*Fineness curve of cement No. 406 as obtained from three analyzers, B, C, and D. (See Fig. 18)*

It is also observed that analyzer *D* would give a better distribution of its separations if the 0.003-inch nozzle were enlarged so as to increase the corresponding fraction by 5 or 6 per cent of the total cement. It is a mere coincidence that the sizes of separation of analyzers *C* and *D* are very close together, in consequence of which the fractions obtained with them are in very close agreement. The points obtained from analyzer *B* are subject to greater uncertainty, owing to the impossibility of obtain-

ing pure slides for calibration. The results of observations giving the data for the curve in Fig. 20 are recorded in Table 6.

TABLE 6

Data Obtained in the Mechanical Analysis of Cement No. 406, from Three Analyzers. (See Fig 18)

Analyzer	Grade	Diameters of average particles				Per cent of total cement
		P	S	G	Average diameter	
	Inch	Inch	Inch	Inch	Inch	
B	0.003	0.00233	0.00258	0.00256		63.10
B	.003	.00229	.00260	.00258		63.20
Mean		.00231	.00259	.00257	0.00249	63.15
D	.003	.00212	.00203	.00211		58.30
D	.003	.00217	.00226	.00225		58.14
Mean		.00214	.00214	.00218	.00215	58.22
B	.002	.00139	.00144	.00138		45.38
B	.002	.00148	.00139	.00149		45.76
Mean		.00144	.00142	.00144	.00143	45.57
C	.002	.00138	.00140	.00144		49.78
C	.002	.00150	.00156	.00163		49.34
Mean		.00144	.00148	.00154	.00149	49.56
D	.002	.00142	.00152	.00151		50.52
D	.002	.00150	.00152	.00148		50.22
Mean		.00146	.00152	.00150	.00149	50.37
C	.001	.00073	.00076	.00073		30.72
C	.001	.00070	.00077	.00079		30.99
Mean		.00072	.00076	.00076	.00075	30.86
D	.001	.00076	.00081	.00077		30.21
D	.001	.00072	.00080	.00072		30.66
Mean		.00074	.00080	.00074	.00076	30.44

Summarizing, briefly, the results of these studies on particle measurements, we have shown that it is possible to calibrate the analyzer simply by determining its sizes of separation, without limiting in any way the dimensions or the operating conditions of the apparatus. The sizes of separation may be determined and

expressed in various ways, that which has been adopted being apparently the simplest and most direct, though not giving the true size as this term is ordinarily interpreted. Should it be found desirable to approximate quite closely the true sizes of separation, two methods of doing this, aside from actually making the three dimensional measurements, have been suggested—first, an approximate reduction factor has been determined by which the true sizes may be computed from the routine measurements; second, the mean true diameter of a set of particles representing the size of separation is very approximately the mean breadth of these particles, a measurement which can be made without difficulty, but less readily than the routine method of measuring diameters without regard to the orientation of the particles. It should finally be emphasized that the success of this routine method of calibration depends primarily on obtaining representative slides of the greatest possible purity, and the analyzer must be so constructed that pure slides can be readily obtained.

VIII. A STUDY OF CONDITIONS WHICH MAY INFLUENCE THE SEPARATIONS OF THE AIR ANALYZER

As stated in the preceding section, a critical study of the possible sources of error in air analysis of cements is a long investigation in itself, and up to the present time only the more obvious conditions which might affect the separations have been investigated. It is intended that the effects of these conditions and their relation to the theory of air elutriation shall be further studied.

The factors or conditions which are most likely to affect the air separations may be included in three groups—(1) those relating to the construction of the analyzer, (2) atmospheric conditions, (3) variations in the quantity and character of the cement. Under the first group will be considered the influence of the shape and dimensions of the apparatus and the effects of abrasion. The second group will include a discussion of external atmospheric conditions, variations in the working pressure, etc. The third group includes a great variety of conditions, comparatively few of which have been thus far investigated. This group is the most important and at the same time the most difficult to cover

thoroughly, and the results to be presented merely indicate what degree of reliability may be placed in the analyses when carried out under the assumption that all normal cements are subject to partition on the same basis.

1. CONDITIONS INVOLVED IN THE CONSTRUCTION OF THE ANALYZER

(*a*) Shape of Bulb, Diameters of Nozzles, etc.—Variations in these parts of the apparatus have already been discussed on pages 27–29. So far as we may judge from experience, these items do not affect the separations, provided their arrangement permits the air stream to have continued or intermittent access to the entire sample of cement. Practically, the shape of the bulb does have an important relation to the amount of cement that can be handled with the minimum air stream, and indirectly, therefore, determines to some extent the minimum size of separation obtainable with a given diameter of stack. The diameters of the nozzles do not need to be accurately determined or specified. It has been our custom to use three nozzles whose diameters are approximately in the ratio of 1:2:3, the smallest being so chosen that the size of its separation is somewhere near the limit of the "impalpable powder." From the dimensions and working pressure of our present apparatus it is possible to compute the approximate size of nozzle for any desired separation in any apparatus of this type.

(*b*) Diameter of Stack.—It was stated on page 27 that the stack used in our present form of apparatus (6.8 cm internal diameter) is probably quite close to the most efficient size to use for three well-distributed separations in the portion of normal cement passing the No. 200 sieve. The reason for this lies in the fact that any particular analyzer of this type is better adapted to one particular size of separation than it is to either finer or coarser separations. Thus, in order to avoid unnecessary abrasion of the cement particles by the action of the air stream, the energy of the latter should be only great enough to insure the proper circulation of the cement. In a given apparatus with a given working pressure this determines the air delivery and consequently the velocity of air up the stack, which in turn determines the size of separation.

Greater air delivery gives a larger size of separation but more abrasion. Smaller air delivery will not handle the cement properly. Hence, we are obliged to design an analyzer which is best adapted to the smaller sizes of separation desired, at the same time permitting the desired range in sizes to be attained without excessive abrasion. In our apparatus the diameter of the stack is practically a minimum for the 0.001-inch grade, and as will be shown under the discussion of abrasion effects, nearly a maximum for the 0.003-inch grade.

(*c*) Length of Stack.—Since the separation of finely divided materials by elutriation methods depends on the "floating power" of the different particles, we should anticipate that the length of the analyzer stack beyond a certain minimum would not greatly affect the separations. However, it can hardly be presumed that particles just capable of suspension in a given air stream remain stationary. It is much more probable that toward the end of an analysis many particles are oscillating irregularly in all parts of the separating chamber. Consequently, if the air stream is retarded slightly by friction near the wall of the stack, the longer the latter, the more opportunity the particles might have to fall back, and we should therefore anticipate that a long stack would either give slightly smaller percentages than a short stack on a given separation or require a longer time for the completion of the analysis. It is also desirable to have the stack as short as possible for the sake of compactness and convenience in setting up. We therefore carried out a series of analyses in which all operating conditions were kept as constant as possible, but with diminishing lengths of stack. Our present form of analyzer was originally constructed as described in Section IV, except that the original length of the stack was 8 feet instead of 5 feet. Two complete analyses were made of the same carefully mixed cement with this stack, the length was then reduced to 7 feet, and two complete analyses of the cement were again made. This process was repeated on a 6-foot stack and finally on the 5-foot stack. The results of these analyses are given in Tables 7 and 8.

TABLE 7

Results of Observations Made to Determine Effect of Length of Analyzer Stack

Length of stack, in feet	Grade	Test No.	Average diameter of maximum particles	Per cent of total cement	Time of analysis
	Inch		Inch		Minutes
8	0.001	1	0.00092	29.46	101
8	.001	2	94	29.79	112
Mean			.00093	29.62	106
8	.002	1	.00187	50.88	140
8	.002	2	193	50.96	144
Mean			.00190	50.92	142
8	.003	1	.00285	59.90	90
8	.003	2	286	59.10	89
Mean			.00286	59.50	90
7	.001	1	.00094	29.61	111
7	.001	2	95	29.73	112
Mean			.00094	29.67	112
7	.002	1	.00188	50.42	140
7	.002	2	.00182	50.48	130
Mean			.00185	50.45	135
7	.003	1	.00282	59.10	113
7	.003	2	273	59.20	90
Mean			.00278	59.15	102
6	.001	1	.00098	29.88	97
6	.001	2	100	30.00	98
Mean			.00099	29.94	98
6	.002	1	.00186	50.94	130
6	.002	2	187	50.90	130
Mean			.00186	50.92	130
6	.003	1	.00267	59.00	101
6	.003	2	276	58.92	93
Mean			.00272	58.96	97
5	.001	1	.00095	30.21	95
5	.001	2	100	30.66	104
Mean			.00098	30.44	100

TABLE 7—Continued

Results of Observations Made to Determine Effects of Length of Analyzer Stack—Continued

Length of stack, in feet	Grade	Test No.	Average diameter of maximum particles	Per cent of total cement	Time of analysis
	Inch		Inch		Minutes
5	.002	1	.00181	50.52	116
5	.002	2	183	50.22	120
Mean			.00182	50.37	118
5	.003	1	.00272	58.30	90
5	.003	2	269	58.14	90
Mean			.00270	58.22	90

In Table 7 the individual results of the two independent analyses are given in order to show the general agreement of repeated separations and calibrations. All the results are given exactly as obtained, and it will be noted that in only one case do the separations fail to agree witihin a few tenths of 1 per cent. The calibrations were made according to the original system of measuring maximum particles and are in fair agreement. Each recorded diameter is the mean of 120 determinations, 40 by each of three observers.

TABLE 8

Summary of Results of Observations to Determine Effect of Length of Analyzer Stack

Length of stack in feet	0.001-inch grade			0.002-inch grade			0.003-inch grade		
	Size of particles	Per cent of total cement	Time of analysis	Size of particles	Per cent of total cement	Time of analysis	Size of particles	Per cent of total cement	Time of analysis
	Inch		Min.	Inch		Min.	Inch		Min.
8	0.00093	29.6	106	0.00190	50.9	142	0.00286	59.5	90
7	94	29.7	112	185	50.4	135	278	59.2	102
6	99	29.9	98	186	50.9	130	272	59.0	97
5	98	30.4	100	182	50.4	118	272	58.2	90

The summary in Table 8 enables us to draw the general conclusion that the length of the analyzer stack beyond a certain minimum does not greatly affect the mechanical analyses. There seems to be a tendency for the 0.001-inch particles to increase in

size with diminishing stack, but the percentage of this grade increases at the same time. On the other hand, the 0.003-inch particles appear to decrease in size with diminishing stack, the percentage of this grade also diminishing. The time required for separations are irregular and may depend more on atmospheric or other conditions than upon the length of stack. Possibly the analyses are completed a little sooner with the shorter lengths. It is intended later to continue this series beyond the minimum permissible length of stack. At present we are unable to account for the slight apparent changes, and are not convinced that further observations will confirm these changes.

(*d*) Abrasion Effects.—It will have been inferred from the description of the analyzer and its operation that the air delivery varies approximately as the square of the size of separation, and consequently the energy of the air stream varies in much the same manner. Now, the greater the energy of the air stream the greater the abrasion of the particles in the residue, and we are therefore interested in determining the actual effect of abrasion in the coarsest separation. We have attempted to determine the effects of abrasion in two ways—first, by blowing a sample with the 0.003-inch nozzle for many hours and noting the final rate of loss; second, by carrying an analysis through the successive grades on a single sample of cement, and comparing these results with analyses made in the usual way, in which fresh samples are used for each separation. The rates of loss during successive hours in the 0.003-inch separation of a 50 g sample of normal cement were as follows:

	Grams
Loss during third hour	0.76
Loss during fourth hour	.42
Loss during fifth hour	.23
Loss during sixth hour	.24
Loss during seventh hour	.22
Loss during eighth hour	.17
Loss during ninth hour	.15

From the ninth to eleventh hours the loss was sensibly constant, and we may assume the value of 0.15 g, or 0.3 per cent, as the loss per hour due to abrasion. In the second test described above, the first analysis was performed on fresh samples; the second was carried out by blowing the 0.001-inch residue

with the 0.002-inch nozzle, and finally the 0.002-inch residue with the 0.003-inch nozzle. The results obtained were as follows:

	0.001 inch	0.002 inch	0.003 inch
Per cent blown from fresh samples	30.84	51.10	58.56
Per cent blown from single sample	30.68	51.08	59.82

These results have been confirmed by other determinations. The total time required for the complete analysis on the single sample was six hours, indicating an average rate of loss of about 0.2 per cent per hour. This rate is, of course, lower than it would be for the 0.003-inch blow alone and roughly confirms that obtained by the other method. The actual time required for the 0.003-inch separation on a normal sample is about 1½ hours, and the maximum loss due to abrasion is therefore probably not greater than 0.5 per cent. For ordinary purposes this loss can be neglected, but allowance can be made for it if desired.

2. ATMOSPHERIC CONDITIONS

(*a*) Temperature and Barometric Conditions.—No certain effects on the separations due to variations in atmospheric pressure and temperature have thus far been detected, and if these have been at all appreciable their effects have been attributed to errors of observation. These conditions enter the problem only in their relation to the density and viscosity of the air, as the working pressure is maintained at a fixed point above atmospheric pressure, regardless of the actual barometric height. Theoretically, the density enters chiefly to alter the velocity of the air delivered by the nozzles, while the viscosity enters chiefly in the carrying power of the air. The maximum effect on the separations would probably be observed when the barometer was low and the thermometer high, but no observations have been made as yet for the definite purpose of establishing this relation.

(*b*) Humidity.—Comparative analyses with the air analyzer have been made under widely varying conditions of humidity, and the results have indicated that there is no necessity for the use of moisture absorbers in the air supply. In fact, if the air is very dry, or if close to the saturation point, the analyzer does not

function as well as it does under normal atmospheric conditions. In the former case there is a noticeable electrical effect in the bulb, the dust adhering strongly to the glass, and the analysis generally requiring a longer time. When the humidity is very high there is more sticking of the dust in the upper part of the stack. Neither of these conditions, however, interfere seriously with the working of the analyzer, nor do they appear to affect the results appreciably.

(*c*) Variations in the Pressure of the Air Supply.—Theoretically, the velocity of air from the nozzles varies as the square root of the pressure in the reservoir. For small variations in pressure, therefore, we should expect the corresponding variations in the air stream to be about one-half as great. But the size of separation varies approximately as the square root of the air velocity, hence we should anticipate that small changes in pressure in the reservoir would have a negligible effect on the separations. This has been confirmed experimentally by making comparative analyses at normal pressure, at 5 per cent below normal, and at 5 per cent above normal. The following percentages of "flour" were obtained from a normal cement under these conditions:

	Per cent.
Pressure 5 per cent above normal	30. 51
Pressure normal	29. 94
Pressure 5 per cent below normal	29. 67

These figures show that a difference of 10 per cent in pressure produced only about 1 per cent difference in the finest fraction. It is therefore quite safe to assume that small variations in pressure may be neglected in the operation of the analyzer.

(*d*) Eddy Currents, Impulses, and Lack of Uniform Velocity in the Stack.—The observations recorded in Tables 7 and 8 furnish the only information available at the present time on possible effects arising from the general turbulence in the bulb and lower part of the separating chamber. It is presumed that pulses and eddy currents gradually disappear as the air stream rises in the separating chamber, and that the impulses imparted to coarse grains of the residue are not sufficient to carry excessively large particles into the collector. Friction of the air stream at the wall of the stack may conceivably produce a slightly higher velocity at the center, but the observations are not conclusive on this point.

3. VARIATIONS IN THE QUANTITY AND CHARACTER OF THE CEMENT

(*a*) Size of Sample—A number of comparable analyses have been made to determine whether the size of sample has any effect upon the apparent percentage value of the various fractions. Differences might be looked for in view of the arbitrary method of determining the stopping points, which in our work are indicated by a rate of loss of 0.02 g per minute for the standard 50 g sample. The following results were obtained with analyzers C and D (see Fig. 18):

Analyzer	Cement	Grade	Size of sample	Per cent of total cement	Time of analysis
		Inch	Grams		Minutes
D	x	0.001	50.00	30.54	151
D	x	.001	33.33	30.42	100
C	y	.001	50.00	34.72	110
C	y	.001	33.33	34.68	76
C	z	.001	50.00	30.90	95
C	z	.001	33.33	30.84	68
C	z	.001	25.00	30.72	55
C	z	.002	50.00	50.62	97
C	z	.002	25.00	50.80	57

The foregoing results indicate a very slight and negligible tendency to obtain higher percentages with the larger samples. The end points in all these analyses were determined by the same rate of loss, and the agreement was quite unexpected before these and similar observations demonstrated that for all practical purposes analyses of samples ranging from 25 to 50 g would give concordant results with exactly the same criterion for completion. It may be noted, however, that the time of the analysis is roughly proportional to the size of sample in a given analyzer, and for different samples the more finely ground material requires a longer time. If samples of the same size are used for the three separations in a given analyzer, the times decrease with increasing size of separation. For example, in analyzer D the complete 0.001-inch separation of a 50-g sample requires normally 150 minutes, the 0.002-inch separation about 120 minutes, and the 0.003-inch separation about 90 minutes. Below are given comparative results of "flour" determinations on small samples of

different sizes in a bulb formerly used with analyzer D but later broken.

Size of sample in grams	Per cent of total cement	Time of analysis
		Minutes
25.00	27.44	100
20.00	27.95	70
16.67	29.34	65
12.50	28.72	46
10.00	28.60	36
8.33	28.56	30

These results are in very fair agreement for the amounts used, the last three in particular agreeing much more closely than the conditions warrant. The variation in the first three analyses is more normal and, in general, the variation is greater for smaller samples.

(*b*) Specific Gravity.—The varying specific gravity is undoubtedly the most important item to be considered in relation to its effect on the analyzer separations. Theoretically, the size of separation varies inversely as the square root of the specific gravity, other conditions remaining unchanged; and as we must deal with a range of at least 10 per cent in the specific gravity of various sized particles, we may expect to find corresponding discrepancies of 5 per cent in the sizes of separation. The varied specific gravity of different cements and of the various sizes of particles of a nonhomogeneous material like cement is an absolutely unavoidable source of error in every elutriation process, but, if necessary, calibrations can be made in important cases, these calibrations requiring but little more time than the actual analyses as now carried out. Many observations on cements of different gravities will be required to establish with certainty the limitations of the analyzer in this respect; that is, to establish the probable range in sizes of separation of any one grade. At present the available data simply indicate what order of error may arise if this and other variables are not taken into account.

The cements from which the data of Tables 1 to 5 have been collected were chosen particularly because they represent a con-

siderable variation in physical characteristics. The pertinent characteristics of four of these cements and the results of the analyzer separations and calibrations are given in the following table:

TABLE 9

Results of Analyzer Separations and Calibrations on Cements of Different Specific Gravities

Cement	Grade	Original condition				After treatment			
		Specific gravity	Ignition loss	Size of separation	Per cent of total cement	Specific gravity	Ignition loss	Size of separation	Per cent of total cement
	Inch		Per ct.	Inch			Per ct.	Inch	
3008	0.001	3.00	5.54	0.00078	33.54	(?)[a]	0.46	0.00082	32.19
	.002			.00146	54.26			.00158	52.60
	.003			.00208	63.02			.00228	61.48
3296	.001	3.13	2.97	.00076	29.55	(?)[a]	0.26	.00078	27.24
	.002			.00151	47.48			.00160	46.32
	.003			.00201	55.46			.00218	54.42
3457	.001	3.24	0.62	.00071	28.80	3.22	1.22	.00076	28.26
	.002			.00144	47.90			.00156	47.96
	.003			.00208	56.94			.00215	56.62
3693	.001	3.12	2.12	.00076	28.74	3.10	3.04	.00078	27.93
	.002			.00162	46.74			.00164	45.78
	.003			.00223	54.30			.00220	53.84

[a] Unfortunately these samples were accidentally destroyed before the specific gravities were redetermined.

Cement No. 3008 has abnormally low gravity and high ignition loss, and fails to meet the ordinary specifications. It appears to be an old sample, or else to have been unduly exposed. Cement No. 3457 has high gravity and low loss, and appears to be fresh from the mill. Cements Nos. 3296 and 3693 are more nearly normal, the latter being ditinguished by certain peculiarities in manufacture. All the cements were tested in the analyzer in their original condition, and calibrations of the separations were made. Portions of Nos. 3008 and 3296 were ignited at about 900° C and again tested and calibrated. Portions of Nos. 3457 and 3693 were exposed to the atmosphere for about six weeks, during which time the average humidity was fairly high, and again tested and calibrated. The results of these retests are given in the last four columns of Table 9.

It was anticipated that the tests of the ignited samples would show lower percentages and smaller sizes of separation than the original samples, especially in the 0.001-inch grade. The percentages of the various fractions are seen to be lower, but the sizes of separation are apparently higher. This must be attributed to the blasted condition of the particles whose edges were so serrated after the heat treatment that accurate measurements could not be made under the microscope. It was also anticipated that the tests of the exposed samples would show higher percentages and larger sizes of separation than the original samples, especially in the 0.001-inch grade. The sizes of separation are seen to be slightly higher, but the percentages are generally lower. This must be attributed to the damp condition of the samples when retested, which probably prevented clean separations in the analyzer. We are not wholly satisfied with this explanation, however, for the 0.003-inch particles appear under the microscope to be only very slightly affected by either treatment. It would undoubtedly be safer at the present time to disregard the results on the treated samples, simply on the ground that all of them were tested in a condition not encountered in normal tests.

Referring to the original tests, it is to be noted that the sizes of separation of the 0.001-inch grade vary inversely as the average specific gravities. In general this is as it should be, for undoubtedly the large differences in gravity are to be attributed to the condition of the finest particles and not of the cement as a whole. Hence, no definite relation can be sought between the average specific gravity of a cement and the sizes of its coarser particles; in fact, the only proper method of studying this problem is to compare the true sizes of separation with the gravities of the separate fractions. Even this is likely to lead to error in the "flour" fraction, of which the average gravity may be appreciably less than that of the largest particles of the fraction. The labor of carrying out this investigation in the manner suggested is great, and perhaps it is better for the present to accept the theoretical effects of gravity as the actual effects than to try to establish them experimentally.

The figures in the fifth column of Table 9 show a range of 10 per cent or more in the corresponding sizes of separation.

In view of the fact that the cements chosen cover nearly the normal range of specific gravity we may assume that this 10 per cent range represents approximately the limits of uncertainty in the size of separation with a given nozzle. This means that the sizes of separation may vary in extreme cases about 5 per cent from the nominal sizes, and by referring to the typical fineness curve in Fig. 20 it is possible to determine what error may be expected in estimating the percentage of any given fraction from its size of separation. If one does this on a carefully plotted curve, he will find that an error of 5 per cent in the size of separation yields an uncertainty of less than 1.5 per cent in the corresponding fraction, expressed as per cent of total cement.

(*c*) AGING.—The effects of aging on the physical properties of cement are presumably the lowering of the specific gravity by absorption of water and carbon dioxide, and an increase in fineness due to possible decrepitation of coarser particles. No tests have been undertaken with the air analyzer to establish these changes directly on given samples of cement, but it is quite certain that the finer fractions especially will show a small increase on account of the gradual changes in gravity.

It has been generally assumed that cements increase in fineness with age, but in certain cases we have established beyond question that cements may decrease in fineness, as indicated by very careful No. 200-sieve tests. This phenomenon can be explained on the assumption of a gradually developing adhesion of very fine material to the coarser particles, sufficient to more than offset any decrepitation that may have taken place. We believe the air analyzer is capable of giving more definite information on this subject, and a series of analyses will be undertaken for this purpose.

(*d*) SHAPE OF PARTICLES.—It has been suggested that the shapes of fine particles might distinguish cements ground in different types of finishing mills, and might possibly have some influence on the air separations. In our earlier microscopic measurements no particular attention was given to this matter, and if there were characteristic differences in the appearance of certain cements they were not sufficiently striking to suggest more careful examination. Recently a number of cements from different mills have been examined with a view to identification simply by the appearance of the

particles as obtained for calibration of the analyzer. These cements were as follows: No. 946, a tube-mill product; No. 1149, tube mill with cyl-peb compartments; No. 1150, tube mill exclusively with steel slugs; No. 2079, a Fuller-Lehigh product; No. 2387, crushed between rolls and air separated; No. 3296, wet-process cement, grinder unknown.

Slides were prepared representing the three sizes of separation of each cement, 18 in all. These were examined individually by three observers, and notes were made of any peculiarities. Some days later attempts to identify the cements were made by each observer, judging solely from the notes he had previously made. In 18 trials, 6 by each observer, 8 were successful and 10 were failures. All the cements were incorrectly identified in at least one instance, notwithstanding the fact that each observer was allowed to study the slide and his notes as long as he desired. In such an examination as this the observer is unconsciously influenced by a number of factors which should not be taken into consideration; for example, color, density, and purity of the slides, and it is believed that the number of failures would have been even greater if new slides had been prepared for the final test. Nevertheless, certain slight differences in the appearance of the different cements were observed. Thus, Nos. 1149 and 1150 were ground from the same material and could not be identified one from the other, although readily distinguished from all the others by the more jagged particles and lack of uniformity in size. Of the remaining cements, Nos. 946 and 3296 possibly showed slightly distinguishing characteristics, but in general it may be stated that the particles of the different mill products do not show well-marked peculiarities.

IX. APPLICATIONS OF THE AIR ANALYZER

1. ROUTINE MECHANICAL ANALYSES OF DIFFERENT BRANDS OF CEMENT

When the air analyzer had been developed to a satisfactory working condition, a series of routine analyses of different cements was undertaken. Between 40 and 50 different brands have been examined to date, and others are being added to the list as opportunity offers in the course of the regular work.

2. COMPARISON OF THE PRODUCTS FROM VARIOUS TYPES OF FINISHING MILLS

One of the practical uses of the analyzer is its adaptation to the study of the products of different types of grinding machinery. Comparative examinations of this sort can be made without calibration of the analyzer, and from examination of a large number of cements a fairly correct notion may be obtained of the characteristics of different mills. For the most reliable comparisons, however, the tests should be made on cements ground from the same clinker, and we have utilized every opportunity to obtain such comparisons. Similarly, the degree of pulverization of different clinkers in a given mill can be determined, and the effects of hardness and other variables can be studied with the aid of the analyzer more satisfactorily than with the aid of sieves alone.

3. STANDARDIZATION OF REGROUND AND AIR-SEPARATED CEMENTS WHICH ARE TO BE USED IN THE FURTHER INVESTIGATION OF THE VALUE OF FINE GRINDING

One of the most important functions of the air analyzer, at least so far as investigative work is concerned, is that of determining the real fineness of cements in a region which no mechanical sieve can reach. It is believed that this apparatus, or some other means of determining extreme fineness, is a prime necessity in an investigation of the value of fine grinding, and cements are now being prepared in the laboratory by regrinding and by continuous air separation, in tests of which mechanical sieves are of little or no value.

4. ADAPTATION OF THE ANALYZER TO OTHER MATERIALS AND ITS LIMITATIONS IN THIS RESPECT

Experience has shown that the analyzer is equally well adapted to separations of other materials than cement, and in many cases may give more consistent results on other materials. For example, excellent separations have been made of ground quartz, emery, alumina, and other hard-grained materials. A new field of usefulness has recently been found in the testing of molding sands, in

which the ordinary clay and silt determinations are especially important but ordinarily made by crude washing and settling methods. In fact, an investigation of molding sands has just been undertaken by the Bureau in which the analyzer will be used. On the other hand, the present form of analyzer has failed in attempts to separate hydrated lime and certain paint pigments, in which the coarse material appears to consist of compact agglomerates of fine particles, or of soft grains. Thus, attempts to analyze a sample of hydrated lime led to the interesting result that the amount of the 0.001-inch grade was apparently greater than that which passed the No. 200 sieve. Results of the same character were obtained in the separation of red lead, and to a less extent in separations of litharge. These peculiarities lead one to consider more carefully just what is meant by the fineness of such materials. In the present analyzer the abrasion or attrition of particles under the air stream is considerable, sufficient in the case of hydrated lime and red lead to easily reduce a large portion of the coarse material to fine particles, whereas the abrasion which takes place on the No. 200 sieve is comparatively small. The action of the Thompson classifier is also very gentle, and certain pigment separations made with it [10] are apparently open to the same criticism as the sieve tests of such materials, in that the agglomerates are not sufficiently broken up. The question arising in cases of this sort is simply, What are to be considered the ultimate particles in a fineness test; or, in other words, how vigorous an action may be permitted in making the proposed separations? So far as our present analyzer is concerned, the reliability of the separations can readily be determined by continuing the maximum separation to such a point that the rate of loss is appreciably constant. If this rate is high as compared with that for cement, it may be assumed that the treatment is too vigorous for accurate analyses, but if of the same order, then the analyses may be considered satisfactory.

[10] See report of Subcommittee J of Committee D-1 on the Testing of White Paints, Proc. A. S. T. M., 13, pp. 406-447; 1913.

X. UNCOMPLETED PHASES OF THE INVESTIGATION

The rather extensive study of No. 200 sieves, the results of which were published in Technologic Papers Nos. 29 and 42 of the Bureau of Standards,[11] may be considered the first step in a general fineness investigation of cements undertaken by the Bureau about three years ago. The development of the air analyzer to a satisfactory working condition marks the practical completion of the second stage of the investigation. Many things remain to be done with the analyzer, however, not the least important of which is to get it into the hands of other investigators who will eventually determine whether the apparatus can be adapted to routine cement testing or whether it will serve only for special and investigative work; but before new analyzers of this type are constructed, it is desirable that the present form be simplified and made as compact as possible. It is anticipated that these improvements can be made, and work in this connection is under way.

As already intimated, a more critical study of the theory of the analyzer, of possible factors which influence the separations, and of improved methods of calibration will be made as opportunity offers. In addition to these lines of work, three others have suggested themselves as worthy of investigation. The most important of these is the construction of an apparatus capable of still further subdividing the 0.001-inch grade or "flour." As seen by Fig. 12, the flour consists of particles varying from a certain maximum down to vanishing size, and constitutes normally some 30 per cent of the total cement. The actual course of the mechanical analysis curve below this point is unknown, and for some purposes it is important to know whether this portion has a double curvature. A single division somewhere near the middle of this fraction might furnish the desired information. It has been assumed, however, that the upper limit of the 0.001-inch grade is low enough to be well within the hydraulically active material, and for the contemplated investigation of finely ground cements the present line of division will probably answer all purposes. The further sub-

[11] Technologic Paper No. 29, Variations in Results of Sieving with Standard Cement Sieves. Technologic Paper No. 42, The Standardization of No. 200 Cement Sieves.

division of this grade is interesting, particularly in relation to other materials than cement. For example, the Bureau has recently been requested to cooperate with other Government departments in developing a standard "dust" composed of organic material. Such materials will probably present the same difficulties in separation as hydrated lime and certain paint pigments, for which it has been shown that the present form of analyzer is not adapted. It is believed, however, that the analyzer can be modified in such manner as to make these fine separations, and, incidentally, the accomplishment of this result would insure a very much more complete analysis of cement than is now possible.

Another matter which promises to facilitate the standardization of the analyzer and improve the uniformity of results to be obtained from different analyzers is the possible use of some inert material of proper specific gravity for calibrating the separations of the different nozzles. The nominal sizes of separation now have to be determined from observations on several supposedly normal cements, which is not only a laborious process but likely to be in error from unsuspected peculiarities. If some inert, finely ground material of definite physical properties approaching cement in specific gravity and granular condition could be discovered, it could undoubtedly be used to advantage for calibration purposes. The fact that such a material would not give precisely the same results as cement particles would probably be more than offset by the uniformity to be obtained.

The remaining item of interest in connection with the analyzer is the possibility of checking up the three-dimensional measurements by collecting and weighing a known number of particles representing the different sizes of separation and computing the average diameter from a knowledge of the specific gravity. The chief incentive for this undertaking is to make possible the comparison of actual and theoretical separations, which is important in its bearing on the proper method of expressing the sizes of separation. The three-dimensional measurements are not made with great accuracy and the deduction of the so-called true diameters by two entirely independent methods would perhaps shed further light on the apparent increase in reduction factors for the finer grades, as shown in Table 5. The particles of the

0.003-inch grade would, of course, be easier to deal with than the finer particles, and a brief computation will indicate the magnitude of the problem. The average true diameter of the 0.003-inch separation is shown to be approximately 0.002 inch or 0.005 cm. The weight of an average particle is the product of volume and specific gravity, or approximately

$$W=\frac{\pi}{6}(0.005)^3 \times 3.2 = 0.0000002 \text{ g}$$

Assuming the practical limit of weighing to be 0.000001 g., it would require 500 of these particles for an accuracy in weighing of 1 per cent. This would therefore be a comparatively easy experiment to carry out, although this degree of accuracy might not be attained. In the case of the 0.001-inch separation, the weight of an average particle would be of the order of one-thirtieth as great as that computed above, and therefore some 15 000 particles would be required to yield an accuracy of 1 per cent in weighing. It remains to be seen whether the counting and weighing of particles of these dimensions can be done with sufficient accuracy to make the attempt to check the microscopic determinations worth while.

XI. SUMMARY

Summarizing briefly this preliminary report on the air analyzer, we may call attention to the following points of chief importance:

1. The design of a satisfactory elutriator is based on certain fundamental requirements, which have been enumerated. If these requirements are embodied in the construction of a given elutriator, its shape, dimensions, and working conditions are of secondary importance, and can be readily adapted to give the desired separations.

2. A review of earlier types of elutriators used or proposed for cement testing shows that some are faulty in design or unnecessarily complicated, while others are probably capable of making good separations. Only one serious attempt appears to have been made to identify the separations in terms independent of the particular apparatus employed, and this not in a manner to enable comparisons to be made with results obtained by others.

3. After a series of preliminary experiments an air analyzer has been developed at the Bureau of Standards which seems to meet the fundamental requirements and appears to give reliable separations of cement and certain other materials. The distinguishing features of the apparatus are: The introduction of an unretarded air stream into a conical-shaped bulb in such manner that the sample under examination is completely and continuously exposed to the action of the air; a separating chamber of considerable height which permits no lodgment of either fine or coarse particles; a tapping device to minimize the adherence of fine dust to any part of the apparatus; and a collector which catches and holds all material passing through the separating chamber.

4. Believing that the main objection to elutriation methods has been the lack of standardization of apparatus heretofore used, we have endeavored to define the separations obtained by the air analyzer entirely in terms of the limiting sizes of particles in the different fractions. These limiting sizes, designated as the "sizes of separation," may be interpreted in different ways, depending upon the methods of measurement. Several methods of measurement have been studied, and a system has been provisionally adopted, which appears to be the simplest and most direct, although not giving directly the "true diameters" of the particles under examination. The relations between the "apparent" and "true" sizes of particles have been expressed by means of reduction factors, which enable one to convert from one system to another.

5. A preliminary study has been made of the effects of abrasion, specific gravity, atmospheric conditions, and other factors on the separations, but much more work remains to be done before the limitations of the analyzer can be established with certainty. It seems probable that under normal operating conditions the nominal sizes of separation of the analyzer determined by calibrations with normal cements will be within about 5 per cent of the actual sizes. This gives rise to a possible error of about 1.5 per cent in estimating the quantity of any desired fraction from the mechanical analysis curve. As the error in determining the percentage of any given fraction by actual separation in the analyzer is probably not greater than 0.5 per cent, we may assume that the routine

analyses, without special calibrations, will enable us to determine the quantity of particles below a given size in any normal cement within 2 per cent. If greater accuracy is desired in special comparisons or in other important cases, calibrations can be made on the material under examination without excessive labor.

6. Further lines of investigation have been suggested, particularly in modifying the apparatus to yield still finer subdivisions. In making this change it is proposed to adapt the apparatus, if possible, to separations of organic and other powders which are not capable of withstanding the abrasive action produced by the air blast in the present type of bulb and nozzles.

7. The analyzer has been developed with the particular purpose in view of determining the granulometric composition of very finely ground and air separated cements to be used in a further investigation of the value of fine grinding, which is now under way. It is also adapted in its present form to comparisons of the products of different finishing mills and to the separation and grading of abrasives and other hard-grained materials. In view of the considerable variety of finely divided materials already tested with the analyzer, the apparatus promises to have a much broader field of usefulness than that for which it was originally designed.

In conclusion, it is desired to acknowledge the valuable assistance rendered by Allen R. Green in making the three-dimensional microscopic measurements on a very large number of small particles.

WASHINGTON, April 26, 1915.

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